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Health Effects Research Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
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AMPHIBOLE MINERAL STUDY

To Complement the Ongoing Characterization of Fine Particulate Environmental Contaminants for Biological Experimentation

by

Paul C. Siebert and Colin F. Harwood
IIT Research Institute
10 West 35th Street
Chicago, Illinois 60616

Contract No. 68-02-1687

Project Officer

David L. Coffin
Health Effects Research Laboratory
Research Triangle Park, N.C. 27711

U.S. ENVIRONMENTAL PROTECTION AGENCY
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HEALTH EFFECTS RESEARCH LABORATORY
RESEARCH TRIANGLE PARK, N.C. 27711

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FOREWORD

The many benefits of our modern, developing, industrial society are accompanied by certain hazards. Careful assessment of the relative risk of existing and new man-made environmental hazards is necessary for the establishment of sound regulatory policy. These regulations serve to enhance the quality of our environment in order to promote the public health and welfare and the productive capacity of our Nation's population.

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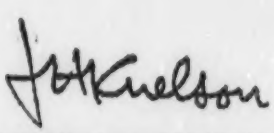
This report details the work and results of a program concerned with the collection of amphibole mineral samples from the Reserve Mining taconite mine in Babbitt, Minnesota. A profile of the ore body was performed by means of petrographic examination of speciments obtained from multiple sites on the rock face. These specimens contained amphibole, magnitite, hornblende, and other minerals. The amphibole fibers were found to have many properties similar to amphibole asbestos types. A large amount of a representative specimen was prepared, blended and placed in vials for animal inoculation.



John H. Knelson, M.D.
Director,
Health Effects Research Laboratory

ABSTRACT

This report details the work and results on a program concerned with the collection of amphibole mineral samples from the Reserve Mining taconite mine in Babbitt, Minnesota and the preparation of highly characterized amphibole fibers from these samples. A profile of the ore body was compiled after a petrographic examination of mineral samples. Various separation and sizing techniques for the fibrous material present in the ore were investigated. Air jet milling of ground and sieved fibrous rock was used to prepare samples for biological experimentation. This material was characterized by optical microscopy, electron microscopy, electron diffraction, x-ray spectroscopy, x-ray diffraction, atomic absorption spectrometry, B.E.T. surface area, and benzene extraction. The fibers were found to have many properties similar to amphibole asbestos types. Vials of weighed amounts of product sample were prepared and purged with nitrogen.

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IITRI personnel contributing to the program were: Dr. Colin F. Harwood, Project Leader; Paul C. Siebert, Principle Investigator; Dr. Anant Samudra and George Yamate, electron microscopists; M. Lal, analytical services; Erdmann Luebcke, sample collection; and Hubert Ashley, technician. The project was administered out of the Fine Particles Research Section, Manager, Mr. J. D. Stockham.

SECTION 1

SUMMARY

This program involved the collection of amphibole mineral samples from Reserve Mining's Peter Mitchell Pit near Babbitt, Minnesota. A geological profile of the ore body was prepared by an examination of ore samples taken from various sites at the mine. Highly characterized amphibole fibers for biological experimentation were prepared from selected mineral samples.

Taconite ore, from which magnetite is extracted, is mined from the Peter Mitchell Pit. The magnetite occurs associated with quartz and various amphiboles, particularly those in the cummingtonite-grunerite series. Amosite is regarded as the fibrous modification of the grunerite (iron rich) end of the series. As amosite asbestos is a health hazard, one of the objectives of this program was to prepare and characterize amphibole fibers for biological experimentation so that it can be determined whether fibers chemically similar to amosite are also a health hazard.

The Peter Mitchell Pit is located at the eastern end of the Mesabi Range in northeastern Minnesota. It has been excavated in the Biwabik Iron Formation of Middle Cambrian age, which trends northeast to southwest and dips gently to the southeast. The formation is subdivided into four members: Lower Cherty, Lower Slaty, Upper Cherty, and Upper Slaty. The pit is mainly in the Upper Cherty, which

is about 140 feet thick, but a little of the Upper Slaty Member is also worked. The formation is truncated east of the pit by the Duluth Gabbro complex. The close proximity of the gabbro is responsible for the high metamorphic grade of the rocks exposed in the pit. The mineralogy of this highgrade zone is distinct from the lower-grade rocks to the west and is characterized by the appearance of amphiboles (grunerite, cummingtonite, hornblende), pyroxenes (hedenbergite, ferrohypersthene), and fayalite.

The pit extends for about eight miles along the strike of the horizon. Nearly 100 specimens were collected on July 1, 1975, from nine localities over a distance of approximately six miles. The specimens, which were selected from broken rock lying at the base of the face and on the quarry floor, include what appeared to average ore types from each locality, but are biased towards samples having layers or masses of iron silicates.

The specimens were examined using a binocular microscope (30x) and 25 thin sections from 24 specimens were prepared and examined by microscope. These 24 specimens were chosen particularly for study of the iron silicate layers and masses, although layers of typical ore are also included in most of the sections. Fibrous to prismatic amphibole, belonging to the grunerite-cummingtonite series, is common and occurs in every thin section. With fine-grained material it is difficult to determine optically whether the amphibole is cummingtonite or grunerite. so this material was referred to as cummingtonite. However, the Mg content of the amphibole has been known to increase toward the contact of the Duluth Gabbro.

Grain size varies considerably. Magnetite occurs as dust, as grains 20-200 μm in diameter, and as large dense aggregate masses and layers. Grunerite-cummingtonite

varies from fibers (less than 1 μm wide and 25-270 μm long), needles (20 μm wide and 170-770 μm long), small blades or prisms (10-40 μm wide and 50-230 μm long), to larger blades (100 μm wide and 2040 μm long). Garnet grades from grains of 50 μm to several centimeters in diameter. Quartz occurs in grains varying from 20-4650 μm. Fayalite varies from approximately 100-1000 μm. Hedenbergite ranges from grains of 50 μm to 2.5 cm. Hornblende, including actinolite, varies from tiny grains (80 μm) to prisms commonly intergrown with cummingtonite (120-870 μm in length) to coarser prismatic grains (5-7 mm long). Ferrohypersthene occurs in irregular or prismatic grains up to 1.6 mm long. Calcite is not common, but is usually coarse-grained (up to 6.0 mm diameter). Biotite is rare and has a maximum length of 290 μm. Magnetite and quartz were found to be abundant; cummingtonite-grunerite and hornblende (actinolite) ranged from sparse to abundant, and calcite from rare to sparse.

An additional 750 lbs. of high fibrous content ore were located and collected on October 2, 1975, for preparation of fibrous material for biological experimentation. Ore containing rich fibrous veins were available only near the incursion of the Duluth Gabbro and occured only in two very localized areas within approximately 100 m of each other.

The fiber separation and sizing techniques that were investigated, in the literature and/or experimentally were:

1. Jaw Crushing
2. Roll Crushing
3. Ball Milling (wet)
4. Air Jet Milling
5. Magnetic Separation
6. Sieving

7. Sedimentation
8. Felvation
9. Serial Filtration by Nuclepore Filters
10. Magnetic Alignment of Fibers
11. Dielectrophoresis
12. Stirred Screening
13. Vertical Spinning
14. Combinations of the Above

Jaw crushing and roll crushing, and mechanical sieving were found to be unnecessary when the material from fibrous veins was used. Wet ball milling could be used to reduce size to the micron and sub-micron ranges; however, this technique introduces the possibility of leaching of the material by the liquid. Air jet milling was found to be successful in first breaking up the bundles into individual fibers and second in reducing the length of the fibers. Magnetic separation would be useful in preparing samples from taconite in that the non-magnetic fraction was more fibrous (85%) than unseparated crushed taconite; however, only a small portion of the crushed taconite was non-magnetic. Sedimentation separated, by diameter, fibrous or non-fibrous particles in fair agreement with theory; however, sedimentation has the disadvantage of not being able to separate fibrous and non-fibrous particles, or by length of fiber, and of the possibility of leaching of the settling material. Felvation has similar disadvantages to sedimentation with the added drawback that it is extremely time consuming and has low productivity due to the low free area and high pressure drop of micron pore sized sieves. Serial filtration by Nuclepore filters could prove useful in separating fibrous and non-fibrous material (of diameter greater than the fiber diam-

eter) however, this technique does not separate by length of fiber and requires aerosolization of a fiber rich sample. Magnetic alignment of fibers was not investigated, as an electromagnet of suitable size and strength was not available and because most amphibole fibers align in two fractions perpendicular to each other. No conclusive separation of fibrous and non-fibrous material was achieved using dielectrophoresis. Stirred screening and vertical spinning have been used with only limited success with chrysotile by Johns-Manville, and thus were not attempted for this material.

Fibers were separated from fiber-rich rocks using several methods. Both hand and vibratory cobbing were used to separate fibrous material (∿1.5kg) in veins. Several rocks were found to consist almost entirely of soft, light green or brown fibrous material. These rocks were crushed, ground, and sieved (<35 mesh) to produce a material (∿3kg) with a high fibrous-to-non-fibrous ratio.

These separated fibrous materials are not necessarily representative in all respects of the majority of the fibers in the ore in the Reserve Mine or in the tailings from the magnetite extraction at Silver Bay, Minnesota. However, this method was used as large quantities of materials with a large fibrous fraction could be produced more easily than by separating fibers from the ore or the tailings.

Jet milling runs, each of 100g of ground and sieved material were successful in producing material that was mostly less than 30 μm in size (length of fiber or diameter of particle) and with a large fibrous fraction. A spinning riffler was used to reduce the sample size to amounts suitable for various types of characterization.

Jet-milled sample #14 was chosen as the product sample after this characterization. This sample was found to be 36.4% (No.) fibrous with 98.5% of the fibers and 99.5% of

the total particles less than 35 μm in major dimension by optical microscopy (400x). The fibers ranged in length from approximately 1.5 to 40.0 μm. Electron microscopy (10,000x) showed this sample to 13.2% fibrous by number. The fibers ranged in length from approximately 0.2 to 32.0 μm, with 99.0% less than 30 μm and 93.9% less than 15 μm. The higher proportion of non-fibrous particles counted by this technique is due to the large number of non-fibrous particles of diameter less than 0.38 μm. Electron diffraction of selected particles showed all of the fibers examined to be amphibole and all of the non-fibrous particles to be crystalline. Chemical analysis by X-ray spectroscopy showed that all of the fibers and non-fibrous particles examined contained magnesium, silicon, and iron. All of the fibers and 64% of the non-fibers contained calcium while 30% of the fibers and 36% of the non-fibers contained manganese. Aluminum and potassium were present in 9% of the non-fibrous particles. X-ray diffraction analysis of the sample showed that cummingtonite, riebeckite and richerite were present. Tremolite and crocidolite were present as trace materials. Atomic absorption spectroscopy was undertaken for manganese, nickel, chromium, and cobalt which were found in amounts of 11.2 mg/g, 224, 34, and 22 μg/g, respectively. The benzene extractables were determined as 2.22 mg/g. The B.E.T. surface area was determined as 34.1 m^2/g.

The electron diffraction results show that the fibers are amphibole. The presence of magnesium, silicon, and especially iron suggest similarity to amosite or crocidolite with some similarity to anthophyllite. The trace quantities of nickel, chromium and cobalt are common to asbestos, while the quantities present suggest anthophyllite as the most likely. The large amount of manganese present is typical of amosite. The X-ray diffraction results indicate the presence of cummingtonite, tremolite-actinolite or crocidolite. The surface area is high for any asbestos type except chrysotile, however, this may be due in part to the size reduction and presence of small non-fibrous particles. Analysis for aluminum,

which was too weak to be detected, and for sodium, which is at the limit of detection for the X-ray spectroscopy equipment used, would be useful in further characterizing the sample.

No definite conclusions can be reached as to the positive identification of this material; however, it has been shown to be similar to different asbestos types with regard to different characteristics.

The sample was reduced in size to amounts of approximately 75 mg which were then put in 30 ml hypo-vials, closed with a resealing silicone rubber septum, purged with nitrogen for at least 10 minutes, and capped with an aluminum seal. Two hundred of these vials have been prepared. The remaining material of this sample and the other separated fibrous material will be stored in case of further requirments at a later date.

SECTION 2

CONCLUSIONS

The Peter Mitchell Pit of the Reserve Mine has been surveyed. The pit is 10 miles in length and about one mile wide at its widest point. It contains a rich variety of minerals including many different amphiboles and pyroxenes, and also magnetite, quartz, fayalite, garnet, calcite, and other mineral species.

Fibrous to prismatic amphiboles pervade the mine and are readily seen in thin sections of rock taken from locations throughout the mine. Fibrous particles are commonly found to belong to the cummingtonite-grunerite series as determined by optical microscopy. As such, they have a variable chemical composition. The chemical structure is further confused by the fact that the cummingtonite-grunerite fibers are commonly intergrown with hornblende and actinolite.

Although numerous methods were considered and tested for the removal of fibers from crushed rock, none were found to be successful. They resulted in a very high (about 90%) proportion of non-fibrous rock. The most satisfactory method of separating out the fibers was to hand-cobb that portion of the rock which was seen to be rich in fibers.

The separated fibers could not be classified using classical techniques such as sedimentation. It was found to be most satisfactory to use an air-jet mill to classify the fibers. The air-jet mill had the combined action of reducing

the fibers to below 30 μm, breaking up fiber bundles and long fibers, and separating out much of the very fine (sub-micron) gangue material.

The fibers collected were characterized using the optical and electron microscopes, x-ray diffraction, atomic absorption, surface area, x-ray probe, and electron diffraction techniques. A benzene extraction was used to determine the oil content of the fiber. The fibers which have been prepared may be regarded as typical of the type of amphibole fiber which may be found in the mine, but they are not representative of the whole mine.

Small, 75 mg portions of the fibers have been accurately weighed into injectable rubber seal vials. They have been purged with nitrogen prior to shipment.

SECTION 3

RECOMMENDATIONS

Extraction of fibers from crushed rock is extremely difficult. It is recommended that the best approach is to hand-cobb fiber-rich rock and then subject it to air-jet milling to break it down.

Fibers are required which are more representative of the fibers which reach the ambient air of Silver Bay. It is recommended that air samples be taken at Silver Bay. Knowing the fiber characteristics to be sought, rock samples containing these fibers may be collected and subjected to the already developed separation methodology.

All fibers or other particulates should be characterized to a degree far above that commonly applied. As a <u>minimum</u>, the fibers should be analyzed by:

- Transmission electron microscope for size and the presence of other particulates
- Selected area electron diffraction
- X-ray probe elemental analysis
- X-ray diffraction
- Surface area
- Pore volume
- Trace element analysis
- Organic extractable content

SECTION 4

INTRODUCTION

Amphibole rocks are very widespread and form the base rock of about one-third of the United States. It has been realized recently that these rocks may contain appreciable quantities of fibers which have a close similarity to commercial asbestos fibers. Only five amphibole minerals are actually given the name asbestos, and they are:

amosite

actinolite

crocidolite

anthophyllite

tremolite

The criteria for inclusion of a fibrous rock in the asbestos list is based on their commercial viability and not on their adverse health threat. To be commercially viable, the fibers have to have a long length to breadth ratio and they have to be easily separated from the ore.

The basis of the defense in the EPA versus Reserve Mining case hinged upon whether the fibers in Reserve's ore tailings was asbestos. Reserve claimed that although the fibers had a similar chemistry to amosite asbestos, they were not amosite asbestos. Strictly, amosite asbestos comes from Penge, South Africa, and takes its name from the Asbestos Mines of South Africa; however, it is a common practice to name all rocks of a particular group after a well

established source of such rocks. Thus, cummingtonite rock is named after Cummington, Massachusetts, although the rock is found in many parts of the world. Reserve also claimed that the fibers were too small to be regarded as asbestos fibers and that there was insufficient medical data for the fibers to be regarded as a health threat.

This program was concerned with the collection of amphibole minerals samples from the Reserve Taconite Mine in Minnesota and the preparation of highly characterized amphibole fibers from these samples. From an examination of ore samples taken from various sites at the mine, a geological profile of the ore body was prepared. This profile was made by a detailed petrographic examination of thin sections prepared from 24 samples. The 24 samples were chosen from the ore samples taken using a binocular microscope at 30-50X.

Fibrous particles were then separated from the fiber-rich rock, reduced in size, and characterized by size, chemistry, electron and x-ray diffraction, surface area, and trace metal and organics content. As the preparation process was dry, there was no problem with leaching of the samples. The separated fibers were then weighed and sealed into hypovials in amounts sufficient for one dose for a group in the biological experimentation, sealed and purged with nitrogen prior to shipment.

SECTION 5

ORE SAMPLE COLLECTION

Taconite ore is mined by the Reserve Mining Company from the Peter Mitchell pit near Babbitt, Minnesota. The magnetite that is extracted occurs associated with quartz and amphiboles in the cummingtonite/grunerite series. The amphibole rocks have the general formula $(MgFe)_7Si_8O_{22}(OH)2$[1]. Cummingtonite is the magnesium rich end member with the theoretical formula $Mg_7Si_8O_{22}(OH)_2$, while grunerite is the iron rich end member with a theoretical formula $Fe_7Si_8O_{22}(OH)_2$.

Amosite is regarded as the fibrous modification of the minerals on the grunerite end of the series; it has a Fe:Fe+Mg ratio of 0.87:1[2]. The chemical composition of several amphibole minerals is given in Table 1. It can be seen that the amosite has a higher Fe_2O_3 content than grunerite and it has been suggested, but not substantiated, that this results from oxidation as a result of igneous activity which is also responsible for recrystallization of grunerite as fibers[3].

The Peter Mitchell pit has been geologically surveyed and has been shown to consist of some 22 layers of rock[4]. The picture is made more difficult by folding, shearing, and faulting throughout the region, and by the presence of an igneous intrusion (the Duluth Gabbro) in the northern part of the pit. However, the area from which the ore is taken consists essentially of three layers. It is upon these layers that this study was concentrated.

Table 1. THE CHEMICAL COMPOSITION OF AMPHIBOLES

	Muscovite	Anthophyllite	Cummingtonite	Amosite (Penge)	Grunerite	Tremolite	Actinolite
SiO_2	46.01	45.50	54.67	49.47	49.01	58.90	55.26
Al_2O_3	35.77	17.64	0.27	0.63	0.00	0.56	2.23
Fe_2O_3	0.83	2.16	0.73	4.15	--	--	1.19
FeO	0.72	9.04	27.16	35.63	44.99	0.22	5.12
MgO	0.33	20.51	13.66	6.57	3.17	24.74	20.41
CaO	0.12	0.87	0.99	0.52	0.31	13.00	12.07
Na_2O	0.76	1.73	0.05	0.02	0.04	0.40	0.59
K_2O	10.42	0.00	0.06	0.20	0.00	0.10	0.10
H_2O	4.31	2.12	1.27	2.40	1.59	1.19	1.81
TiO_2	0.14	0.23	0.03	0.25	0.05	0.02	0.04
Cl	--	0.05	--	--	--	--	--
F	0.38	0.02	0.07	--	1.00	0.30	0.31
P_2O_5	0.12	--	--	--	0.10	--	0.01
Cr_2O_3	--	--	--	--	--	--	0.32
NiO	--	--	--	--	--	--	--
MnO	0.04	0.16	0.80	0.61	0.37	0.42	0.31
Total	99.95	100.03	99.76	100.45	100.63	99.85	99.77

On July 1, 1975, more than 100 samples weighing some 300 lbs were collected from the Peter Mitchell Pit at Babbitt, Minnesota by Drs. Harwood and Woodland and Mr. Luebcke. The samples for geological examination were collected at widely different sites within the mine. Samples of the rock tailings after the removal of magnetite at the Reserve Mining plant at Silver Bay, Minnesota, were also collected. Large quantities of samples were taken from the northeast corner of the mine, near the Duluth Gabbro, where veins containing obvious fibers were located. These samples were collected for testing of methods of separating and sizing of fibrous material.

On October 2, 1975, approximately 750 lbs of high fibrous content ore were located and collected. It was found that the ore containing rich fibrous veins was a very localized phenomenon. Such samples were available only near the incursion of the Duluth Gabbro and occurred only in two very localized areas within approximately 100 m of each other. Several samples were also collected of "typical" taconite ore and cummingtonite, chosen by Reserve Mining's geologist who was assisting in the sample collection.

SECTION 6

ORE BODY PROFILE

The Peter Mitchell Pit is located near Babbitt, at the eastern end of the Mesabi Range in northeastern Minnesota. It has been excavated in the Biwabik Iron Formation of Middle Cambrian age. The Iron Formation trends northeast to southwest and dips gently to the southeast. It is subdivided into four members: Lower Cherty, Lower Slaty, Upper Cherty, and Upper Slaty. The pit is mainly in the Upper Cherty, which is about 149 feet thick, but a little of the Upper Slaty Member is also worked. The Iron Formation is truncated east of the pit by the Duluth Gabbro complex, the contact of which swings away from the top of the Biwabik Formation and then approximately parallels it to the southwest at a distance of about one to one and a half miles; south of Mesaba it turns sharply southward away from the Iron Formation.

The close proximity of the Gabbro is responsible for the high metamorphic grade of the rocks exposed in the pit and also approximately southwestward to the vicinity of Mesaba. The mineralogy of this highgrade zone is distinct from the lower-grade rocks to the west, and is characterized by the appearance of amphiboles (grunerite, cummingtonite, hornblende), pyroxenes (hedenbergite, ferrohypersthene), and fayalite [5,6].

The pit extends for about eight miles along the strike of the ore horizon, and samples were collected on July 1, 1975, from nine localities over a distance of a little more

than six miles. The working face of each bench is approximately 35 feet high, that is, over a quarter of the thickness of the total exploited thickness of ore beds. Nearly 100 specimens varying in size from about three inches on a side to over six inches were collected. The specimens were selected from broken rock lying at the base of the face and on the quarry floor. They include what appeared to be average ore types from each locality, but are biased towards samples having layers or masses of iron silicates.

GENERAL MICROSCOPIC DESCRIPTION

A total of 25 thin sections from 24 specimens were prepared and examined. These specimens were chosen particularly for study of the iron silicate layers and masses, although layers of typical ore are also included in most of the sections.

Fibrous to prismatic amphibole is common and occurs in every thin section; it belongs to the grunerite-cummingtonite solid solution series. The division between grunerite $[(Fe^{2+}Mg)_7Si_8O_{22}(OH)_2]$, the iron-rich end of the series, and cummingtonite, $[(MgFe^{2+})mSi_8O_{22}(OH)_2]$ is arbitrary, as there is no natural gap in the series. It is commonly taken that 70% or more of the Fe^{2+} molecule is called grunerite and, conversely, that more than 30% of the Mg molecule is cummingtonite[7]. Optically this approximates to the change of 2V from negative (grunerite) to positive (cummingtonite). With fine-grained material, however, it is difficult to determine; in this report the amphibole of this series is referred to as cummingtonite, although some may actually be grunerite. The Mg content of the amphibole increases towards the contact of the Duluth Gabbro. French (1968) indicates that it is cummingtonite that is present within one and a quarter to one and a half miles of the contact, and grunerite beyond. The Mitchell Pit's collecting localities A and B

lie within this distance, and localities C, D, and E just beyond (see Figure 1).

Grain Size

Grain size varies considerably. Magnetite occurs as dust, as grains 0.02-0.2 mm in diameter, and up to large, dense aggregate masses and layers. Grunerite-cummingtonite has considerable variation in both grain size and habit, from fibers (25 μm long and less than 1 μm wide; or longer -- up to 0.08-0.27 mm), needles (0.17-0.77 mm long and 0.02 mm wide), small blades or prisms (0.05 mm long and 0.01 mm wide up to 0.23 mm long and 0.04 mm wide), to larger blades (2.04 mm long and 0.1 mm wide).

Garnet grades from grains about 0.05 mm in diameter to masses several centimeters across. Quartz occurs in very fine grains (0.02 mm), medium-sized grains (0.4-0.8 mm), and very coarse (up to 4.65 mm in some sections). Fayalite varies from approximately 0.1-0.96 mm.

Hedenbergite, similarly, grades from very fine grains (0.05 mm) through medium grains (0.03-3.3 mm) to coarse grains (9.3 mm) and very coarse (2.5 cm). Hornblende, including actinolite, varies from tiny grains (0.08 mm) to prisms commonly intergrown with cummingtonite (0.12-0.87 mm in length) and to much coarser prismatic grains 5.0-7.0 mm long. Ferrohypersthene occurs in irregular or prismatic grains up to 1.63 mm long.

Calcite is not common, but is usually coarse-grained (up to at least 6.0 mm in diameter). Biotite is rare; maximum grain size is about 0.29 mm in length.

LITHOLOGIC AND MICROSCOPIC DESCRIPTIONS

The following is a brief lithologic description of the specimens based on their examination under a binocular microscope (X30), together with a microscopic description of the thin sections.

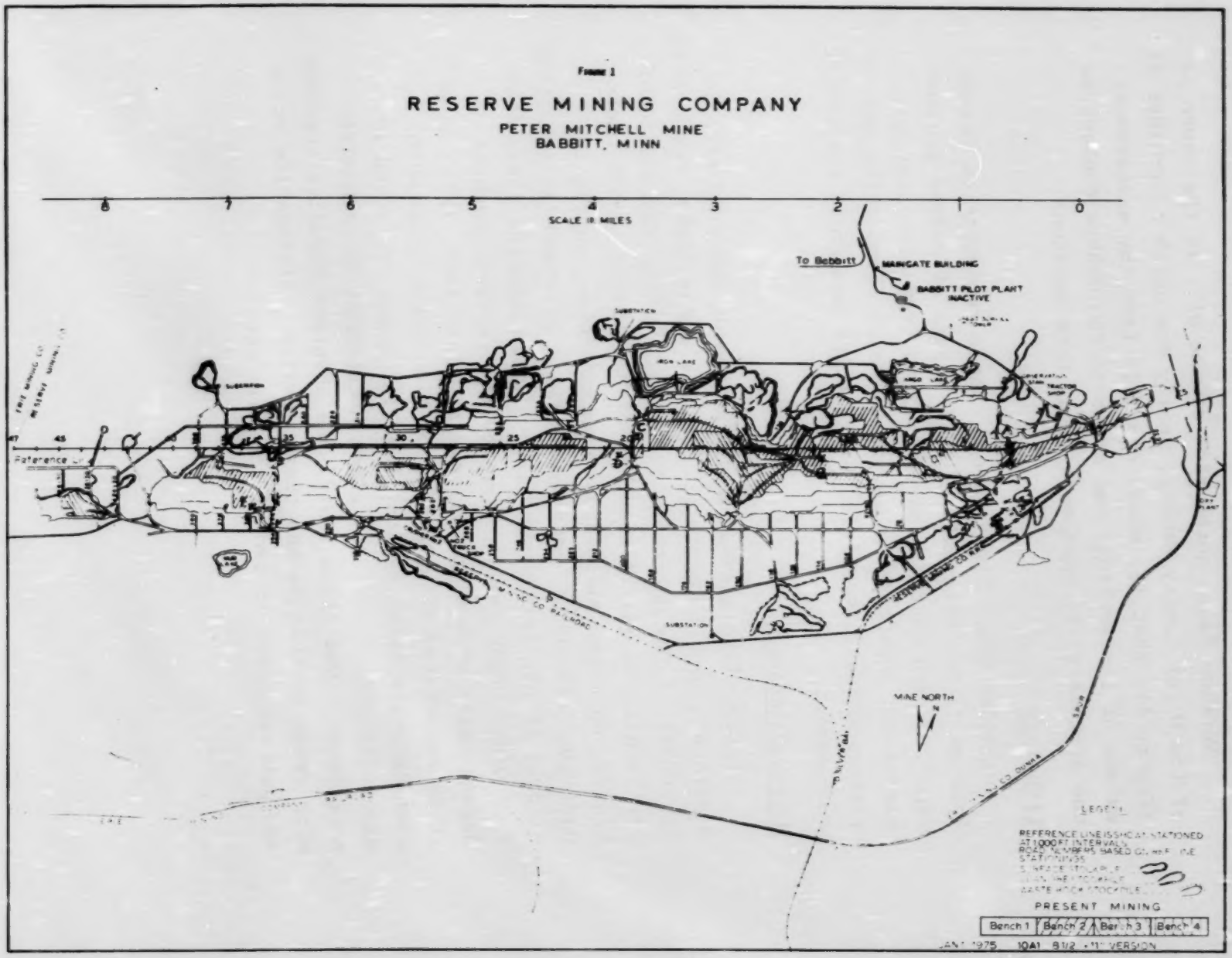


FIGURE 1
RESERVE MINING COMPANY
PETER MITCHELL MINE
BABBITT, MINN
8 7 6 5 4 3 2 1 0
SCALE IN MILES
To Babbitt
MANGATE BUILDING
BABBITT PILOT PLANT
INACTIVE
SUBSTATION
IRON LAKE
Reference
SUBSTATION
MINE NORTH
LEGEND
PRESENT MINING
Bench 1
Bench 2
Bench 3
Bench 4
JAN. 1975 10A1 8 1/2 x 11" VERSION

Locality A. N.

Working face, north side of the pit, in the lower part of the Upper Cherty Member, nearly due north of crusher #1 (Figure 1), that is, about one mile from the easternmost extent of the workings and close to the Gabbro contact to the southeast; 11 specimens, four thin sections.

Lithology -

Banded ore: fine-grained, dense magnetite in layers 4-10 mm thick, intercalated with layers of coarse-grained magnetite and quartz; coarse-grained iron silicates with quartz and magnetite in layers up to 50-60 mm thick and in masses; garnet, amphibole, and fibrous amphibole associated with garnet.

Micro-lithology -

Dense, fine-grained magnetite bands and band rich in quartz and magnetite grains. In one slide (AN8), the granule structure of the magnetite is evident within the mosaic of recrystallized quartz together with a little garnet, hornblende, calcite, and fibrous cummingtonite, partly in radiating clusters. Ferrohypersthene is common in two other slides; it has fringes of fibrous cummingtonite (less than 2 μm wide) which partly replaces the ferrohypersthene. Hedenbergite occurs in the same sections (AN3, AN7); in AN7 it is associated with patches of blue-green hornblende. Cummingtonite, with typical lamellar twins, is found in magnetite-rich bands, and green hornblende in magnetite-poor bands. One slide (AN12) has abundant fayalite together with green hornblende and cummingtonite. Hisingerite veins and patches occur in two of the thin sections.

Locality A. S.

Working face, due south of Locality A. N. and towards the upper part of the Upper Cherty Member, the closest locality to the Gabbro; 28 specimens, four thin sections (three specimens).

Lithology -

Six of the specimens are from irregular sheets or veins composed of quartz and matted fibrous amphibole (identified microscopically as cummingtonite-grunerite) stained by iron oxide from weathering. These sheeted veins apparently cut the ore layers in irregular fashion and are relatively common at this locality. The banded ore is typical: fine- and medium-grained quartz and magnetite in bands varying from 5-100 mm thick; prismatic amphibole prominent in some magnetite-rich layers; iron silicate layers and masses with garnet, epidote, biotite, and prismatic amphibole identified in some specimens; calcite vein in one specimen. A prominent constituent of some specimens is fibrous amphibole; it occurs in association with magnetite, quartz, and other iron silicates as veins cutting magnetite-quartz layers and in masses on and penetrating magnetite layers.

Micro-lithology -

Cummingtonite is common in all four sections; in one (AS14c), it is abundant, prismatic to fibrous in habit, in part yellow-brown due to alteration, and intergrown with hornblende; magnetite is disseminated throughout. In another slide (AS17ca), magnetite-rich bands have lenses of quartz with acicular fibrous masses of cummingtonite, and other layers have abundant light green hornblende (or actinolite); plagioclase occurs sparingly. The other two thin sections (AS17cb; AS20c) are rich in magnetite, have common acicular to prismatic cummingtonite, coarse

plagioclase and biotite, full of tiny grains or dusty magnetite, and quartz.

Locality Bi

Working face, in the upper middle portion of the Upper Cherty Member, 1.5 miles west of Locality A (the 11,500 foot mark on the pit reference line, Figure 1); five specimens, one thin section.

Lithology -

Fine-grained, dense magnetite layers alternate with medium-grained quartz and magnetite; fibrous amphibole masses and layers with other iron silicates in four of the five specimens.

Micro-lithology -

Magnetite shows a granule structure in a quartz mosaic, in part with aggregates of prismatic to fibrous cummingtonite in patches and also needles of cummingtonite in the quartz. There are bands rich in dense magnetite. Aggregates of blue-green hornblende intergrown with cummingtonite are abundant. There is also a little chlorite, calcite, and a vein of yellow-brown hisingerite.

Locality Bii

Working face, near Locality Bi but in the middle part of the Upper Cherty Member; 11 specimens, three thin sections.

Lithology -

Typical ore is composed of alternating dense magnetite-rich layers and medium-grained quartz-magnetite layers; green silicate layers; garnet in some; prismatic pyribole (amphibole or pyroxene) disseminated throughout the quartz-magnetite layers and silicate layers in a number of specimens.

Micro-lithology -

One section (BM26b) has a large mass of garnet and large aggregates of hedenbergite intergrown or altering to acicular to fibrous cummingtonite; fayalite with hedenbergite, prismatic cummingtonite, and a little magnetite; prismatic and fibrous cummingtonite in patches and in part light brown due to alteration.

In the other two slides (BM27b; BM27e), magnetite in granule structure is present; acicular cummingtonite abundant in the granules and also in aggregates and surrounding area with granule structure. One of the sections (BM27b) has a little hedenbergite and hornblende.

Locality C

Working face, in the middle of the Upper Cherty Member, approximately 1.5 miles west of Locality B (about the 19,600 foot mark on the pit reference line, Figure 1); 12 specimens, four thin sections.

Lithology -

Alternating dense magnetite and coarse quartz-magnetite layers the most common; silicate layers include masses of fibrous amphibole in radiating clusters and in seams up to 17 mm thick; calcite with coarse-grained amphibole, quartz, and magnetite.

Micro-lithology -

In section CM28c, magnetite occurs in a granule structure in a mosaic of quartz with abundant needle and fibrous cummingtonite and some pleochroic brown to very dark brown mineral, which is either hisingerite or nontronite; aggregates of hedenbergite have fringes of cummingtonite and are cut by veins of hisingerite.

Section CM28e has a dense aggregate of blue-green hornblende, in part plumose, with fringes of fine needle to fibrous cummingtonite; magnetite has a granule structure, and there are ovoid areas of plagioclase with hornblende surrounding and grown into them and containing acicular cummingtonite.

Section CM28g has both dense magnetite-rich bands and bands with granule structure, the latter with abundant fibers and prismatic grains of cummingtonite. Another band is dense with needle and fibrous cummingtonite in radiating clusters.

The fourth section (CM28d) contains considerable calcite and quartz; acicular cummingtonite occurs in the calcite; hornblende is common and partly intergrown with cummingtonite.

Locality D

Working face, in the upper portion of the Upper Cherty Member, about a quarter of a mile southwest of Locality C (near the 20,500 foot mark on the pit reference line, Figure 1); 10 specimens, three thin sections.

Lithology -

Fine-grained, dense magnetite layers intercalated with fine- to medium-grained silicate layers with calcite, quartz, and magnetite; garnet-rich layers with associated green pyribole; layer with fibrous amphibole in radiating groups; sulfide streaks in some of the dense magnetite layers; hematite in some specimens together with magnetite, quartz, jasper, and stellate fibrous amphibole.

Micro-lithology -

Section DU30c has a zone with magnetite grains and dust in fine-grained quartz; a mass of poikilitic garnet with quartz, a little cummingtonite, brown-colored alteration material, and some hedenbergite altering to brownish-yellow hisingerite.

Section DU30a has, in addition to magnetite-rich masses, quartz, and calcite, abundant hedenbergite with intergrown cummingtonite. The hedenbergite exhibits varying styles of alteration to brown hisingerite. A little blue-green hornblende is also present.

The third section (DU30e) has bands of varying composition; much coarse magnetite with interstitial quartz and abundant fine needles of cummingtonite and green hornblende in some bands; bands and lenticles rich in fibrous cummingtonite; quartz with abundant cummingtonite needles; another band has common green-brown biotite, abundant cummingtonite, some in radiating clusters, and a little chlorite (after biotite?).

Locality Ei

Working face, in the lower middle part of the Upper Cherty Member, about half a mile west of Locality D (about the 22,750 foot mark on the pit reference line, Figure 1); five specimens, two thin sections.

Lithology -

Calcite veins and quartz veins with sulfides cut layers of fine-grained, dense magnetite and coarser layers of quartz and magnetite with iron silicates, including green prismatic and fibrous amphiboles. One specimen has thin layers of green fibrous amphibole.

Micro-lithology -

Section ELM32a has abundant magnetite in dense bands and in bands with granule structure; in the dense bands there are lenses of fibrous or needle cummingtonite and a little green hornblende. There is also a band of fine-grained, green, prismatic hornblende with a little cummingtonite, a band of matted cummingtonite with a little hornblende, magnetite grains, and green clots. A band rich in

magnetite has abundant brown hisingerite (or nontronite?). The granule zone has much quartz, fibrous cummingtonite in stellate bunches, and some hornblende. There is a band of cummingtonite fibers in stellate of plumose structure, and another with much magnetite and a mass of fibrous cummingtonite with green or greenish-brown alteration product of hisingerite. A quartz-rich zone has cummingtonite fibers penetrating it as a fringe.

The other section (ELM32e) has coarse calcite, coarse prismatic hornblende (or actinolite), a little plagioclase, quartz, and a little fibrous cummingtonite; hornblende intergrown with cummingtonite. Aggregates of prismatic cummingtonite occur with patches of cummingtonite fibers, brown-colored because of alteration; some fine-grained cummingtonite has a plumose structure and grades into an area with laths of green hornblende.

Locality Eii

Working face, in the lower part of the Upper Cherty Member, adjacent to Locality Ei; seven specimens, two thin sections.

Lithology -

Fine-grained, dense magnetite with coarse quartz and magnetite, green pyribole, and sulfides; silicate layers with magnetite and prismatic amphibole. One specimen has a mass of fibrous green amphibole, and another has a brown vein of partly radiating fibrous amphibole with quartz. Drusy purple quartz is present on fine-grained magnetite with disseminated green iron silicate.

Micro-lithology -

Section EL33e has bands rich in magnetite grains with interstitial fibrous cummingtonite in some and dark green hornblende in others; other bands are rich in fibrous

amphibole -- an intergrowth of cummingtonite and hornblende -- with a little magnetite. Another band has magnetite, quartz, and needle to fibrous cummingtonite, partly in stellate clusters, and rare hornblende and plagioclase. There is also some brown to green-brown hisingerite (or nontronite?) as an alteration product.

The other section (EL33f) has a magnetite-rich zone with aggregates of intergrown hornblende-cummingtonite needles, quartz with needles of amphibole, blue-green hornblende, and prismatic cummingtonite. There is an area of coarse quartz with prismatic to needle intergrown hornblende-cummingtonite. In another zone, quartz and magnetite occur with needle cummingtonite in stellate patterns and a little hornblende.

Locality F

Working face, in the upper part of the Upper Cherty Member, one and a half miles west of crusher #2, a little over two and a half miles west of Locality E (at the 36,500 foot mark on the pit reference line, Figure 1), and less than two miles from the westernmost limit of the pit (the westernmost collecting locality); nine specimens, two thin sections.

Lithology -

Hematite with jasper and magnetite; garnet conspicous in several of the specimens with green iron silicates; green fibrous amphibole associated with garnet in one specimen; layers rich in fibrous amphibole in another specimen. Alternate layers of fine-grained, dense magnetite and coarser-grained quartz plus magnetite with iron silicates are common with blotches and patches of hematite. Coarse-grained calcite together with garnet, prismatic amphibole, and fine-grained magnetite layers make up another specimen.

Micro-lithology -

Section FU34e has dense magnetite bands with interstitial cummingtonite and a little garnet; abundant needle cummingtonite in matted layers with fine-grained magnetite and common tiny garnet. Some bands with magnetite in granule structure also have cummingtonite, common blue-green hornblende, and a little garnet. Quartz has dense inclusions of fibrous aggregates of cummingtonite.

The other section (FU34a) has a large area of abundant quartz and aggregates of needle to fibrous cummingtonite with "frayed-out" ends; also aggregates of hedenbergite and cummingtonite, the hedenbergite having intergrown cummingtonite; sparse occurrence of fine-grained magnetite in granule structure distributed throughout the quartz mosaic; large garnet, partly anisotropic, has inclusions of hedenbergite and cummingtonite; another large garnet has a fringe of matted cummingtonite fibers with some hedenbergite, a little blue-green hornblende, and a very small amount of calcite, and, in places, a very narrow outer rim of hisingerite. A vein-like aggregate of fibrous cummingtonite and hedenbergite cuts a sinuous path across the slide. The cummingtonite in this thin section has a very pale green color and is, thus, probably iron-rich or grunerite.

SUMMARY OF MINERAL OCCURRENCES

The approximate mineral abundances for the different zones of the Upper Cherty Member and averaged from the 25 thin sections are shown in Table 2. Sample photomicrographs of thin sections are shown in Figure 2.

Table 2. MINERAL OCCURRENCE BASED ON 25 THIN SECTIONS

Zone of Upper Cherty	Magnetite	Quartz	Cummingtonite-Grunerite	Hedenbergite	Ferro-hypersthene	Hornblende (Actinolite)	Garnet	Fayalite	Calcite	Biotite	Plagio-clase
Upper	X	X	X-O	△-O		O-△	X-O		•	•	•
Upper Middle	X	X	O			X			•		
Middle	X	X	X	X		O-△	X	△	△		△
Lower Middle	X	X	O-X			△-O			•		•
Lower	X	X	X-△	O	O	X-△	△	△	•		•

X - abundant

O - common

△ - sparse

• - rare

AS 14e - Aggregate of Acicular to Fibrous Cummingtonite-Grunerite
(Yellow by Alteration; Some Isotropic Yellow-Brown Substance)
A Little Intergrown Hornblende (4x)

BM 27e - Magnetite and Quartz in Granule Structure, with Acicular
to Fine Needle and Fibrous Cummingtonite and Grunerite (4x)

Figure 2. Selected fiber-bearing thin sections

CM 28c - Magnetite and Quartz in Granule Structure; Abundant Needle to Fibrous Cummingtonite-Grunerite, with a little Hedenbergite largely altered to intergrown with Cummingtonite-Grunerite

DU 30e - Dense Aggregate of Matted Needles and Fibers of Cummingtonite-Grunerite, with a fringe of Fibrous, Radiating Cummingtonite-Grunerite; Biotite at lower border (4x)

Figure 2. (continued)

ELM 32a - Stellate Aggregates of Cummingtonite-Grunerite and Hornblende, Set in Quartz Matrix with Magnetite Grains (4x)

Figure 2. (continued)

SECTION 7

FIBROUS SAMPLE PREPARATION

In order to prepare fibrous samples suitable for biological experimentation from the ore samples collected at the Peter Mitchell Pit, Babbitt, Minnesota, several processes were required. The fibrous material had to be separated from the non-fibrous gangue, ground or fractionated into the desired sizes, divided into amounts suitable for use, and sealed into vials. During these processes, the fibrous samples were, as much as possible, to be representative of those fibers present throughout the ore body, unchanged chemically and not contaminated by other substances.

FIBER SEPARATION AND SIZING TECHNIQUES

Many types of fiber separation and sizing techniques and equipment were investigated. Those that were considered are:

1. Jaw Crushing
2. Roll Crushing
3. Ball Milling (wet)
4. Air Jet Milling
5. Magnetic Separation
6. Sieving
7. Sedimentation
8. Felvation

9. Serial Filtration by Nucleopore Filters
10. Magnetic Alignment of Fibers
11. Dielectrophoresis
12. Stirred Screening
13. Vertical Spinning
14. Combinations of the Above

These techniques were investigated in the literature and, if promising, experimentally to determine their applicability to this particular separation and sizing problem. Depending on the success of the various techniques under different operating conditions and whether the ore or the fibrous veins in the ore were used as raw material, a separation and sizing process was to be developed from the above techniques. In the following paragraphs, the above techniques will be discussed.

A jaw crusher is available at IITRI that will accept pieces of rock of 4 in. major diameter and break them to approximately ½ in. pieces. This equipment simulates the operation of industrially used crushing equipment. The product size distribution is also similar to those produced industrially.

A roll crusher is also available at IITRI that will accept the product of the jaw crusher and will break it to 90% less than 10 mesh. This has to be accomplished at various gap settings of the roll crusher alternated with screen sieving. This equipment also simulates the operation and size distribution of industrially used crushers.

A ball mill is available that simulates industrial operation. If used, it would have been operated wet to maximize preservation of the fibers' crystal structure. Almost any size distribution can be attained by controlling the time and conditions of operation.

Air jet milling uses centrifugal forces to break up particles. The particles of the desired size then escape through a cyclone type exit. This type of sizing has been used by Johns-Manville in the preparation of asbestos fiber samples. A Jet Pulverizer Model 04-503 4" Micron-Master air jet mill with a capacity of 30-50 lb/hr at an air flow of 50 scfm at 100 psig was obtained to test its suitability for this type of fibrous sample. The jet mill consists of a 4in. diameter, shallow, circular grinding chamber with four angled nozzles equally spaced around the chamber periphery. These nozzles discharge a high energy fluid, such as air or steam, imparting turbulence and a circular motion. The feed particles are impacted upon each other causing breakage. The fine fragments are carried out with the discharged air and collected in a cyclone. The coarse particles are thrown toward the wall and are further ground. Thus, air jet milling accomplishes size reduction with no contact of the material being ground by any other substance except filtered air. A final filter capable of holding 4-8 in. x 10 in. filters was constructed. Nuclepore 0.8 μm pore size filters were used to maximize the capability of recovering the fine fraction that escapes from the jet mill cyclone. Preliminary test runs showed that the length of almost all the fibers was reduced to less than 200 μm with most of the fibers less than 30 μm.

Magnetic separation is one of the steps used in the industrial processing of taconite to iron ore at the Reserve mine. It is used to separate the iron fraction from the tailings. Examination by optical microscope of minus 325 mesh (< 44 μm) hammer crushed taconite before and after electromagnetic separation showed that the total size fraction was approximately 60% fibrous, the magnetic fraction approximately 20% fibrous, and the non-magnetic fraction approximately 85% fibrous. Thus, magnetic separation would

be a useful step in sample preparation from the raw taconite ore in that it enriches the fibrous fraction and simulates the production of tailings in the actual operation. However, this may have to be done for a larger size fraction as the iron content of the amosite-like fibers may cause them to exhibit magnetic properties when in small sizes. This was somewhat confirmed as the magnetic fraction for the magnetic field used was much larger than the non-magnetic fraction.

Mechanical sieving is used to separate the product after roll crushing and ball milling. The smallest size mesh practical for moderate amounts to be practicable is a 325 mesh (<44 μm). Therefore, this would be an intermedidate step if sample preparation is started with the taconite ore.

Sedimentation may be used to separate particles by their terminal settling velocity. Stoke's Law applies for spherical particles at Reynold's Number $\leq$ 1.0. For fibers settling with their axis normal to their velocity, the relationship is more complex, involving an iteration on the Reynold's Number for Re $\leq$ 0.5. The settling velocities of both spherical particles of the density of cummingtonite (ρ = 3.1 g/cm^3) and fibrous particles of the density of amosite (ρ = 2.8 g/cm^3) have been calculated. The settling velocity of a fiber is greater than that of the same diameter spherical particle and the difference increases as the diameter decreases.

Sedimentation experiments were conducted to determine the accuracy of the theory for the particles involved. Hammer crused taconite of minus 325 mesh was sedimented to take the fine fraction. Theoretically, this fraction should consist of fibers less than 3.3 μm diameter and particles less than 9.6 μm in diameter. This method separated diameter

fairly well both for fibers and particles; however, many non-fibrous particles were present in both the coarse and fine fractions. Some agglomeration took place in the fine fraction during its separation from the fluid. Also, the sedimentation technique is maninly insensitive to fiber length. Sedimentation of hand-cobbed fibers into three size fractions was also investigated. As hand-cobbed fibers contain much less gangue material, further separation is not as critical or necessary. Sedimentation was performed for the theoretical fiber diameters of $D_f \geq 3.3$ μm, 3.3 μm $> D_f \geq 1.0$ μm, and $D_f < 1.0$ μm (equivalent to spherical particle diameters of $D_p \geq 9.6$ μm, 9.6 μm $> D_p \geq 4.5$ μm, and $D_p < 4.5$ μm). Optical microscope examination of the sized fractions showed the actual separation by fiber diameter to be nearly that predicted; however, aspect ratios varied from the minimum of three to nearly fifty. The majority of fibers ranged in aspect ratio from three to ten, but the mass distribution would be greatly skewed by the few fibers of much greater length present. Therefore, sedimentation was shown to be a reasonable method of sizing fibers by diameter in large quantities. However, sedimentation has the disadvantages of not being able to separate by shape or length of fiber and of the possibility of leaching of the settling material by the sedimenting fluid.

Felvation is a technique developed at IITRI for fractionating powders. It was initially developed by Kaye and Jackson[8] and since has been modified by other workers to overcome some of the practical operating difficulties. The technique involves aspects of fluidization, elutriation and sieving to separate powders into size fractions between sieves. This technique has, to our knowledge, never been attempted for fibers. An additional complication is that sieves as small as 10 μm are difficult to obtain. For fibers elutriating with their axis normal to the flow as would

normally be expected for micron size fibers, the cut size for felvation would be the length. Thus, the non-fibrous particles of diamter less than the fiber length required will also pass through the sieve. In the case of a 10 μm sieve, felvation would have approximately the same effect as sedimentation as fibers of length 10.0 μm by 3.3 μm are settled in the same time as 9.6 μm diameter particles. Thus, the fibers can be sized by either of these methods, but only separated from a portion of the non-fibrous particles present. Felvation of the fine sedimented fraction hammer crushed taconite using both Nuclepore filters and 10 μm sieves was investigated. The Nuclepore filters could not withstand the suction necessary to draw the particles through the filter. The sieve method did very little to improve separation of fibers from gangue material or to improve sizing of fibers and was extremely time consiming. Felvation of the finer fractions of sedimented hand-cobbed fibers using a 5 μm sieve was found to be even slower than when a 10 μm sieve was used. The amount of material passing through the sieve was of almost negligible mass, while the degree of separation from the gangue was not appreciable. Therefore, felvation was found to be of very low productivity and very time consuming. However, it might prove of some value in removing the longer fibers or in separating the smaller diameter fibers from the gangue if magnetic alignment of fibers is found to be feasible.

Serial filtration of asbestos fibers by Nuclepore filters is a technique developed by Spurny[9]. This technique uses a 8.0 or 5.0 μm Nuclepore filter to separate the larger particles and a 0.2 μm Nuclepore filter to collect the asbestos fibers. In air, the fibers tend to align with the flow and pass through the larger pore size filter. However, this technique has only been used for separation of fibers from ambient air and does not separate by length of fiber.

The additional problem of aerosolizing a fiber-rich sample for separation is also posed.

The magnetic alignment of asbestos fibers has been investigated by Timbrell[10]. He has found that all types of asbestos fibers of respirable size align in response to a magnetic field when settling in a fluid. Amosite fibers in particular align in two fractions parallel and normal to the field and of generally differing size. Thus, the fibers in sedimentation, felvation, or serial filtration could be aligned for one fraction to be parallel or normal to the flow. This technique could prove of great value in combination with other techniques.

This technique was not investigated as an electromagnetic of suitable size and strength was not available and because amosite fibers align in two fractions perpendicular to each other.

H.A. Pohl[11,12,13] is the major investigator of dielectrophoresis as a technique for separation of particles. Dielectrophoresis employs the effect of a non-uniform electric field on the polarity induced on particles of high dielectric constant. The particle will move toward the stronger region of the non-uniform field. This effect can be utilized to separate particles of differing dielectric constants by setting up an apparatus such that a constant force is put on the particle throughout the non-uniform field. The apparatus is then tilted such that the particles in a fluid flow down the length of the apparatus and the force of the field opposes the gravitational force across the width. Thus, the particles of higher dielectric constant are preferentially taken to the high side of the apparatus. Such an "isomotive cell" was constructed, and some experiments run to test the separation of fibers from rock. No effective separation was achieved with the apparatus as used.

Stirred screening is a method developed with some success by Johns-Manville[14] . The purpose of this technique is to keep the fibers parallel to the screens (10 μm - 44 μm) in order to segregate by length; however, limited success was achieved in that separations could only be made less than 5 μm and greater than 10 μm. There was also some overlap of lengths in the separations. This technique was used on ground chrysotile fibers of industrial purity.

Another possible method is that of spinning a test tube full of fibers in suspension at low speed to separate by length. In preliminary studies with carbon fibers[14] the longer fibers came out over the top of the test tube while the shorter ones remained. The mechanism and parameters involved are unknown, so it is unknown whether this method might be suitable for fractionating asbestos fibers by length.

Many combinations of the above methods are possible, depending on the applicability of the various techniques and whether the starting material is the ore itself or only the fibrous veins. A possible process for the separation and sizing of fibers from the raw ore would involve the following techniques:

1. Jaw Crushing to reduce size
2. Roll Crushing to reduce size
3. Ball Milling (wet) to reduce size
4. Magnetic Separation to remove the magnetic, fiber-lean fraction
5. Sedimentation to remove the larger diameter fibers and particles
6. Serial felvation by Nuclepore filters using magnetic alignment of fibers to separate first by diameter and to remove non-fibrous particles and then by length.

A possible process from the fibrous vein material would involve the following techniques:

1. Air Jet Milling to reduce size and allow only the fines by aerodynamic diameter to escape
2. Serial filtration by Nuclepore filters using magnetic alignment to fractionate by length
3. Sedimentation to fractionate by diameter in each length fraction (2 and 3 could be reversed)

Another possible process would be sedimentation of the tailings material produced at Silver Bay, Minnesota, after the extraction of magnetite from the taconite ore. One of the techniques suggested above would have to be used to separate the fibers from the other gangue material. These sample process give some idea of the complexity of the problem and of the number of techniques and steps that may have to be involved to produce samples of acceptable characterization. These problems are separate from but relate to those of the quantity of sample that can reasonably be produced in a given time from a quantity of ore.

FIBER SEPARATION AND SIZING

Fibers were separated from rocks containing large numbers of fibers in significant veins both by hand and by mechanical vibrator. Both methods were found to bring about a major reduction in the amount of non-fibrous gangue present from the amount present in crushed fiber bearing rock. Size of the fibers was dependent on the method used and whether large agglomerates were further reduced in size mechanically. Approximately 1.5 kg of this material was produced; however the process was very time consuming due to the low bulk density of the material of 0.65g/cc (40.6 lb/ft^3).

Several rocks were found to consist almost entirely of soft, light green or brown fibrous material. Crushing, grinding, and sieving (< 35 mesh) of these rocks produces a

material that has a very high ratio of fibrous to non-fibrous material. Approximately 3 kg of this fine fibrous material was produced. The obviously different types of fibers were kept separate by physical characteristics and separation technique as much as practicable.

These separated fibrous materials are not necessarily representative in all respects of the majority of the fibers present in the ore in the Reserve mine or of the fibers in the tailings from the processing at Silver Bay, Minnesota. However, as these materials consist of mostly fibrous material and could be produced much more easily than by attempting to separate the fibrous and non-fibrous fractions from the ore or the tailings, this method was used to produce a large quantity of material with a large fibrous fraction.

Jet milling was conducted for a series of six 100 g samples of ground and sieved material in order to determine optimum operating conditions. The operating conditions of the tests are given in Table 3. Due to the number of parameters involved and possible problems with sample representativeness, the optimum operating conditions were not readily apparent. However, several of the runs had little or no material larger than 100 μm and most of the material smaller than 10 μm.

It was then decided in a meeting with Dr. Coffin that approximately 15 g of fibrous material of 0-30 μm length would be satisfactory. Therefore, more detailed characterization of the two 100 g samples (No.'s 12 and 14) found most promising by optical microscopy and of the unmilled material was undertaken. For this more extensive characterization, a spinning riffler was used to successively reduce sample sizes for representative analysis. After this characterization, consisting of optical and electron microscopy, X-ray spectroscopy, electron and X-ray diffraction, atomic

Table 3. JET-MILL OPERATING CONDITIONS

Sample No.	Feed Setting	Pressure (psig)	
		Feed	Mill
9	9	50	40
10	9	65	65
11	9	70	75
12	8	40	40
13	8	45	60
14	10	60	65

absorption for some trace elements, B.E.T. surface area, and solvent extraction for organics. After this characterization the "best" material (No. 14) was chosen for biological experimentation. The other fibrous material is being retained in case of further requirements at a later date.

PREPARATION OF SAMPLES FOR SHIPPING

The sample for biological experimentation was then successively reduced in size using a spinning riffler to 200 approximately 75mg representative amounts for shipment. The 75mg amounts were used as that is the amount needed for one dose for a group of animals in the biological experiment. Each representative amount was weighed out to the nearest 0.1mg and placed in a 30ml hypo-vial. Each vial was then sealed with a resealing silicone rubber septum and purged with nitrogen for at least 10 minutes, at a pressure of at least 2 in. Hg. An aluminum seal was then fastened over the top of each vial for shipping.

SECTION 8

CHARACTERIZATION OF FIBROUS SAMPLES

OPTICAL MICROSCOPY

Samples were counted by optical microscope using phase contrast and a Porton graticule at 400 or 625X. The counting procedure used was that of the Joint AIHA-AICGIH Aerosol Hazards Evaluation Committee[15]. In the first series of counts of the six jet-milled samples of the ground and sieved material, at least 100 particles were counted to determine the sizes of the fibrous and non-fibrous fractions and their approximate size distributions. The results of these counts are shown in Table 4. From these results, it was concluded that samples 12 through 14 might be satisfactory for biological experimentation. A spinning riffler was used to reduce the sample to representative portions for further analysis. Counts of at least 100 fibers were then conducted of the unmilled material (1 and 2) and samples 12 and 14 at 400X. The results of these samples (see Table 5) show that the jet-milled samples (12 and 14) have a reduction in the length of fibers(to 100% $\leq$ 70 μm) and the amount of non-fibrous material (from $\sim$ 80-90% to 60-70% non-fibrous). Sample photomicrographs are shown in Figure 3. The jet-mill seems to be effective in breaking up bundles of fibers into individual fibers, reducing their length, and reducing the size of many of the non-fibrous particles so that they are too small to be collected in the cyclone. The results of this analysis suggest the use of sample 14 for biological exper-

Table 4. JET-MILLED SAMPLES PARTICLE COUNTS; OPTICAL MICROSCOPE, 625X

Sample Number	Fibers Counted	Percentage of Particle of Length and Diameter in μm											
		0<L≤10				10<L≤100				L>100			
		D≤1	1<D≤36	D>36	N.F.	D≤1	1<D≤36	D>36	N.F.	D≤1	1<D≤36	D>36	N.F.
9	266	16.1	8.3	0	61.3	4.9	8.6	0	0.8	0	0	0	0
10	234	12.8	6.8	0	54.9	6.0	8.6	0	1.7	0	0.9	0	0
11	261	12.6	10.3	0	64.4	4.6	6.1	1.5	0.4	0	0	0	0
12	267	32.2	9.7	0	46.4	7.1	7.1	0	0.4	0	0	0	0
13	232	35.3	4.3	0	37.1	17.2	4.7	0	1.3	0	0	0	0
14	118	31.4	4.2	0	28.[illegible]	16.9	11.9	0.8	0.8	0	0.8	0	0

N.F. - Non-Fibrous

Table 5. SIZE DISTRIBUTION BY NUMBER OF FIBERS AND NON-FIBERS; OPTICAL MICROSCOPE AT 400X

(a) Sample 1

Diameter (μm)	Length (μm)										
	0.0-0.8	0.8-1.1	1.1-1.5	1.5-2.2	2.2-4.4	4.4-8.8	8.8-17.5	17.5-35.0	35.0-70.0	>70.0	Total
0.0-0.8			2 (1.2%)	20 (12.3%)	18 (11.1%)	7 (4.3%)	2 (1.2%)	2 (1.2%)	0 (0.0%)	0 (0.0%)	51 (31.3%)
0.8-1.1					41 (25.2%)	16 (9.9%)	5 (3.1%)	1 (0.6%)	0 (0.0%)	0 (0.0%)	63 (38.8%)
1.1-1.5					3 (1.8%)	15 (9.3%)	3 (1.8%)	1 (0.6%)	1 (0.6%)	0 (0.0%)	23 (14.1%)
1.5-2.2						12 (7.4%)	3 (1.8%)	2 (1.2%)	0 (0.0%)	0 (0.0%)	17 (10.4%)
2.2-4.4							4 (2.5%)	1 (0.6%)	0 (0.0%)	0 (0.0%)	5 (3.1%)
4.4-8.8								2 (1.2%)	0 (0.0%)	0 (0.0%)	2 (1.2%)
>8.8									0 (0.0%)	1 (0.6%)	1 (0.6%)
Total Fibers	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	2 (1.2%) [0.2%]	20 (12.3%) [2.4%]	62 (38.1%) [7.6%]	50 (30.9%) [6.1%]	17 (10.4%) [2.1%]	9 (5.4%) [1.1%]	1 (0.6%) [0.1%]	1 (0.6%) [0.1%]	162 (99.5%) [19.7%]
Non-Fibrous	185 [22.6%]	198 [24.2%]	132 [16.1%]	78 [9.5%]	49 [6.0%]	14 [1.7%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	656 [80.1%]
Total Particles	185 [22.6%]	198 [24.2%]	134 [16.3%]	98 [11.9%]	111 [13.6%]	64 [7.8%]	17 [2.1%]	9 [1.1%]	1 [0.1%]	1 [0.1%]	818 [99.8%]

(b) Sample 2

Diameter (μm)	Length (μm)										
	0.0-0.8	0.8-1.1	1.1-1.5	1.5-2.2	2.2-4.4	4.4-8.8	8.8-17.5	17.5-35.0	35.0-70.0	>70.0	Total
0.0-0.8			2 (0.7%)	52 (17.9%)	45 (15.5%)	14 (4.8%)	4 (1.4%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	117 (40.3%)
0.8-1.1				3 (1.0%)	62 (21.4%)	22 (7.6%)	9 (3.1%)	2 (0.7%)	0 (0.0%)	1 (0.3%)	99 (34.1%)
1.1-1.5					12 (4.1%)	12 (4.1%)	13 (4.5%)	4 (1.4%)	0 (0.0%)	0 (0.0%)	41 (14.1%)
1.5-2.2						14 (4.8%)	8 (2.7%)	2 (0.7%)	0 (0.0%)	0 (0.0%)	24 (8.2%)
2.2-4.4						2 (0.7%)	3 (1.0%)	2 (0.7%)	1 (0.3%)	0 (0.0%)	8 (2.7%)
4.4-8.8							1 (0.3%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	1 (0.3%)
>8.8								0 (0.0%)	0 (0.0%)	1 (0.3%)	1 (0.3%)
Total Fibers	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	2 (0.7%) [0.1%]	55 (18.9%) [1.8%]	119 (41.0%) [3.9%]	64 (22.0%) [2.1%]	38 (13.0%) [1.2%]	10 (3.5%) [0.3%]	1 (0.3%) [0.0%]	2 (0.6%) [0.1%]	291 (100.0%) [9.5%]
Non-Fibrous	821 [27.0%]	1016 [33.4%]	614 [20.2%]	183 [6.0%]	91 [3.0%]	29 [1.0%]	7 [0.2%]	1 [0.0%]	0 [0.0%]	0 [0.0%]	2762 [90.8%]
Total Particles	821 [27.0%]	1016 [33.4%]	616 [20.3%]	238 [7.8%]	210 [6.9%]	93 [3.1%]	45 [1.4%]	11 [0.3%]	1 [0.3%]	2 [0.1%]	3053 [100.3%]

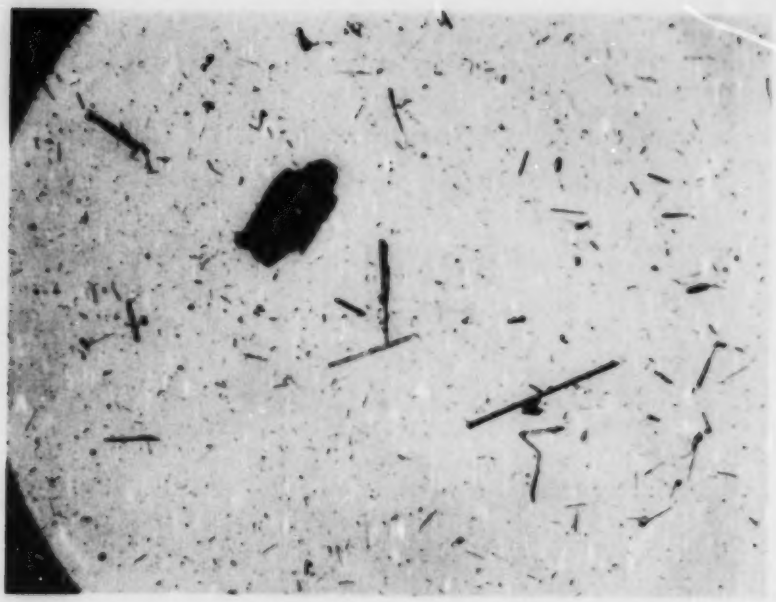
Table 5. (continued)

(c) Sample 12

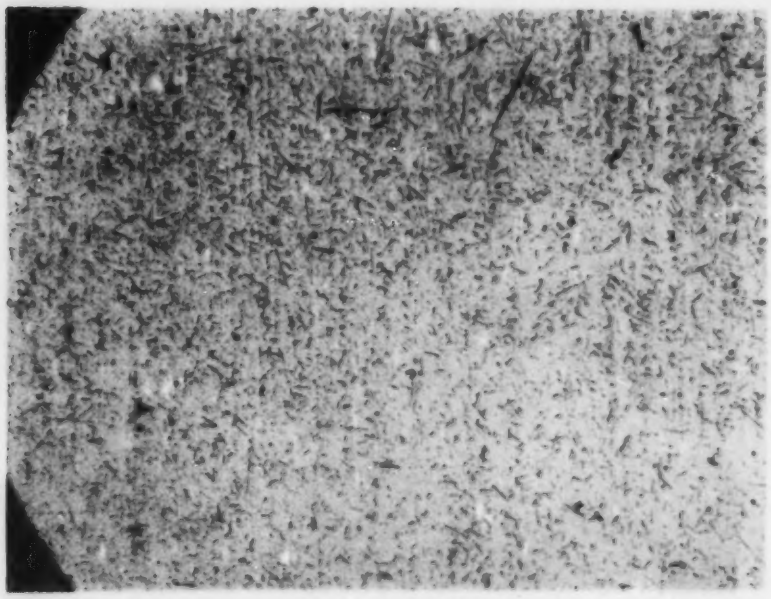
Diameter (μm)	Length (μm)										
	0.0-0.8	0.8-1.1	1.1-1.5	1.5-2.2	2.2-4.4	4.4-8.8	8.8-17.5	17.5-35.0	35.0-70.0	>70.0	Total
0.0-0.8				22 (8.9%)	24 (9.7%)	5 (2.0%)	3 (1.4%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	54 (22.0%)
0.8-1.1					49 (19.9%)	19 (7.7%)	5 (2.0%)	1 (0.4%)	0 (0.0%)	0 (0.0%)	74 (30.0%)
1.1-1.5					14 (5.7%)	14 (5.7%)	16 (6.5%)	6 (2.4%)	1 (0.4%)	0 (0.0%)	51 (20.7%)
1.5-2.2						13 (5.3%)	19 (7.7%)	5 (2.0%)	0 (0.0%)	0 (0.0%)	37 (15.0%)
2.2-4.4						1 (0.4%)	22 (8.9%)	4 (1.6%)	0 (0.0%)	0 (0.0%)	27 (10.9%)
4.4-8.8								1 (0.4%)	1 (0.4%)	0 (0.0%)	2 (0.8%)
>8.8									2 (0.8%)	0 (0.0%)	2 (0.8%)
Total Fibers	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	22 (8.9%) [2.5%]	87 (35.3%) [10.0%]	52 (21.1%) [6.0%]	65 (26.5%) [7.5%]	17 (6.8%) [2.0%]	4 (1.6%) [0.5%]	0 (0.0%) [0.0%]	247 (100.2%) [28.5%]
Non-Fibrous	73 [8.4%]	291 [33.6%]	122 [14.1%]	76 [8.8%]	41 [4.7%]	13 [1.5%]	4 [0.5%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	620 [71.6%]
Total Particles	73 [8.4%]	291 [33.6%]	122 [14.1%]	98 [11.3%]	128 [14.7%]	65 [7.5%]	69 [8.0%]	17 [2.0%]	4 [0.5%]	0 [0.0%]	867 [100.1%]

(d) Sample 14

Diameter (μm)	Length (μm)										
	0.0-0.8	0.8-1.1	1.1-1.5	1.5-2.2	2.2-4.4	4.4-8.8	8.8-17.5	17.5-35.0	35.0-70.0	>70.0	Total
0.0-0.8				9 (2.5%)	20 (5.6%)	7 (2.0%)	2 (0.6%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	38 (10.7%)
0.8-1.1				3 (0.8%)	61 (17.2%)	49 (13.8%)	30 (8.5%)	6 (1.7%)	1 (0.3%)	0 (0.0%)	150 (42.3%)
1.1-1.5					22 (6.2%)	27 (7.6%)	23 (6.5%)	3 (0.8%)	0 (0.0%)	0 (0.0%)	75 (21.1%)
1.5-2.2						29 (8.2%)	21 (5.9%)	11 (3.1%)	2 (0.6%)	0 (0.0%)	63 (17.8%)
2.2-4.4						6 (1.7%)	17 (4.8%)	3 (0.8%)	1 (0.3%)	0 (0.0%)	27 (7.6%)
4.4-8.8							0 (0.0%)	0 (0.0%)	1 (0.3%)	0 (0.0%)	1 (0.3%)
>8.8								0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
Total Fibers	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	12 (3.3%) [1.2%]	103 (29.0%) [10.6%]	118 (33.3%) [12.1%]	93 (26.3%) [9.6%]	23 (6.4%) [2.4%]	5 (1.5%) [0.5%]	0 (0.0%) [0.0%]	354 (99.8%) [36.4%]
Non-Fibrous	36 [3.7%]	216 [22.2%]	194 [20.0%]	88 [9.1%]	61 [6.3%]	20 [2.1%]	2 [0.2%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	617 [63.6%]
Total Particles	36 [3.7%]	216 [22.2%]	194 [20.0%]	100 [10.3%]	164 [16.9%]	138 [14.2%]	95 [9.8%]	23 [2.4%]	5 [0.5%]	0 [0.0%]	971 [100.0%]

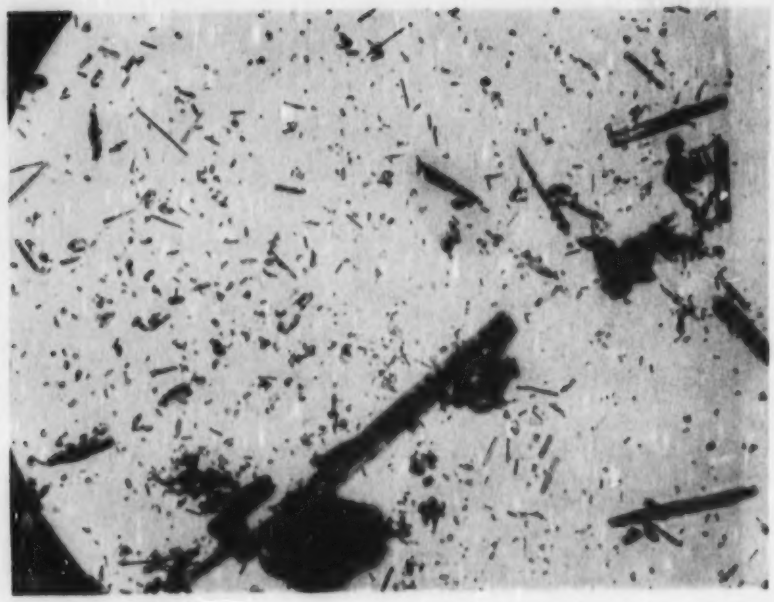


(a) Unmilled Ground & Sieved Material (Sample #2; 65X)



(b) Milled Ground & Sieved Material (Sample #14; 65X)

Figure 3. Optical microscope photomicrographs



(c) Unmilled Ground & Sieved Material (Sample #1; 407X)

(d) Milled Ground & Sieved Material (Sample #14; 407X)

Figure 3. (continued)

imentation as this sample has the least amount of fibers greater than 35 μm in length (1-6%) and greater than 4.4 μm in diameter (0.3%).

ELECTRON MICROSCOPY

Samples were sized by electron microscopy using the JEOL-100C electron microscope in the tramsmission mode. Counts were made at 10,000X. Approximately 100 particles including at least 10 fibers were counted to determine the relative numbers of fibrous and non-fibrous particles. Approximately 100 fibers were then counted to determine their size distribution. The results of these counts are given in Table 6. Sample photomicrographs are shown in Figure 4. These counts of the four ground and sieved samples show that at least 85% of the particles are non-fibrous and consist largely of particles less than 0.38 μm in diameter, which are not visible at 400X. However, the results show again that sample 14 has greater size reduction than sample 12. (It should be noted that many visibly large particles did not remain suspended in the unmilled samples (1 and 2) when the suspended sample was taken to place on the electron microscope grid).

ELECTRON DIFFRACTION

Electron diffraction was also accomplished using the JEOL 100C. Early analysis showed all of the fibers both from fibrous veins in the ore and from the ore itself were amphibole in structure. As shown in Table 7, all of the fibers in the four analyzed samples were also found to be crystalline. Of the 43 non-fibrous particles analyzed in the four samples only one was found to be non-crystalline. Therefore, both the fibrous and non-fibrous material is almost totally crystalline. Samples of electron diffraction patterns are shown in Figure 5.

Table 6. SIZE DISTRIBUTION BY NUMBER OF FIBERS AND NON-FIBERS; TRANSMISSION ELECTRON MICROSCOPE AT 10,000X

(a) Sample 1 -- Fiber and Non-Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	6 (6.1%)	16 (16.2%)	10 (10.1%)	3 (3.0%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	36 (36.4%)
0.12-0.25		9 (9.1%)	11 (11.1%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	20 (20.2%)
0.25-0.50			14 (14.1%)	4 (4.0%)	6 (6.1%)	0 (0.0%)	1 (1.0%)	0 (0.0%)	25 (25.2%)
0.50-1.00				4 (4.0%)	2 (2.0%)	2 (2.0%)	0 (0.0%)	0 (0.0%)	8 (8.0%)
1.00-1.50					4 (4.0%)	1 (1.0%)	1 (1.0%)	0 (0.0%)	6 (6.0%)
1.50-2.50					3 (3.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	3 (3.0%)
>2.50						1 (1.0%)	0 (0.0%)	0 (0.0%)	1 (1.0%)
Total Fibers	6 (6.1%) [0.5%]	25 (25.3%) [2.1%]	35 (35.3%) [3.0%]	11 (11.0%) [0.9%]	16 (16.1%) [1.4%]	4 (4.0%) [0.3%]	2 (2.0%) [0.2%]	0 (0.0%) [0.0%]	99 (99.8%) [8.4%]
Non-Fibrous	808 [69.0%]	164 [14.0%]	86 [7.3%]	8 [0.7%]	5 [0.4%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	1071 [91.4%]
Total Particles	814 [69.5%]	189 [16.1%]	121 [10.3%]	19 [1.6%]	21 [1.8%]	4 [0.3%]	2 [0.2%]	0 [0.0%]	1170 [99.8%]

Table 6. (continued)

(b) Sample 2 -- Fiber vs. Non-Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	1 (8.3%)	3 (25.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	4 (33.3%)
0.12-0.25		1 (8.3%)	3 (25.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	4 (33.3%)
0.25-0.50			1 (8.3%)	0 (0.0%)	1 (8.3%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	2 (16.6%)
0.50-1.00				0 (0.0%)	0 (0.0%)	2 (16.7%)	0 (0.0%)	0 (0.0%)	2 (16.7%)
1.00-1.50				0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
1.50-2.50					0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
>2.50						0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
Total Fibers	1 (8.3%) [1.1%]	4 (33.3%) [4.4%]	4 (33.3%) [4.4%]	0 (0.0%) [0.0%]	1 (8.3%) [1.1%]	2 (16.7%) [2.2%]	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	12 (99.9%) [13.2%]
Non-Fibrous	57 [62.7%]	13 [14.3%]	6 [6.6%]	2 [2.2%]	0 [0.0%]	1 [1.1%]	0 [0.0%]	0 [0.0%]	79 [86.9%]
Total Particles	58 [63.8%]	17 [18.7%]	10 [11.0%]	2 [2.2%]	1 [1.1%]	3 [3.3%]	0 [0.0%]	0 [0.0%]	91 [100.1%]

(c) Sample 2 -- Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	0 (0.0%)	8 (8.1%)	8 (8.1%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	17 (17.2%)
0.12-0.25		4 (4.0%)	8 (8.1%)	4 (4.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	16 (16.1%)
0.25-0.50			11 (11.1%)	10 (10.1%)	7 (7.1%)	7 (7.1%)	0 (0.0%)	0 (0.0%)	35 (35.4%)
0.50-1.00				6 (6.1%)	8 (8.1%)	2 (2.0%)	1 (1.0%)	0 (0.0%)	17 (17.2%)
1.00-1.50					4 (4.0%)	5 (5.0%)	1 (1.0%)	1 (1.0%)	11 (11.0%)
1.50-2.50					1 (1.0%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	2 (2.0%)
>2.50						1 (1.0%)	0 (0.0%)	0 (0.0%)	1 (1.0%)
Total Fibers	0 (0.0%)	12 (12.1%)	27 (27.3%)	21 (21.2%)	20 (20.2%)	16 (16.1%)	2 (2.0%)	1 (1.0%)	99 (99.9%)
Non-Fibrous									
Total Particles									

Table 6. (continued)

(d) Sample 12 -- Fiber vs. Non-Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	0 (0.0%)	0 (0.0%)	2 (14.3%)	1 (7.1%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	3 (21.4%)
0.12-0.25		0 (0.0%)	0 (0.0%)	2 (14.3%)	1 (7.1%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	3 (21.4%)
0.25-0.50			2 (14.3%)	1 (7.1%)	0 (0.0%)	0 (0.0%)	1 (7.1%)	0 (0.0%)	4 (28.5%)
0.50-1.00				1 (7.1%)	2 (14.3%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	3 (21.4%)
1.00-1.50					1 (7.1%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	1 (7.1%)
1.50-2.50						0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
>2.50						0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
Total Fibers	0 (0.0%) [0.0%]	0 (0.0%) [0.0%]	4 (28.6%) [4.5%]	5 (35.6%) [5.6%]	4 (28.5%) [4.5%]	0 (0.0%) [0.0%]	1 (7.1%) [1.1%]	0 (0.0%) [0.0%]	14 (99.8%) [15.7%]
Non-Fibrous	39 [43.8%]	20 [22.5%]	13 [14.6%]	2 [2.2%]	1 [1.1%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	75 [84.2%]
Total Particles	39 [43.8%]	20 [22.5%]	17 [19.1%]	7 [7.8%]	5 [5.6%]	0 [0.0%]	1 [1.1%]	0 [0.0%]	89 [99.9%]

(e) Sample 12 -- Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	1 (1.0%)	2 (2.0%)	5 (5.0%)	1 (1.0%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	10 (10.0%)
0.12-0.25		3 (3.0%)	12 (12.0%)	2 (2.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	17 (17.0%)
0.25-0.50			6 (6.0%)	7 (7.0%)	2 (2.0%)	3 (3.0%)	0 (0.0%)	0 (0.0%)	18 (18.0%)
0.50-1.00				3 (3.0%)	14 (14.0%)	8 (8.0%)	4 (4.0%)	0 (0.0%)	29 (29.0%)
1.00-1.50					4 (4.0%)	3 (3.0%)	4 (4.0%)	1 (1.0%)	12 (12.0%)
1.50-2.50						5 (5.0%)	2 (2.0%)	3 (3.0%)	10 (10.0%)
>2.50						2 (2.0%)	1 (1.0%)	1 (1.0%)	4 (4.0%)
Total Fibers	1 (1.0%)	5 (5.0%)	23 (23.0%)	13 (13.0%)	21 (21.0%)	21 (21.0%)	11 (11.0%)	5 (5.0%)	100 (100.0%)
Non-Fibrous									
Total Particles									

Table 6. (continued)

(f) Sample 14 -- Fiber vs. Non-Fiber Distribution

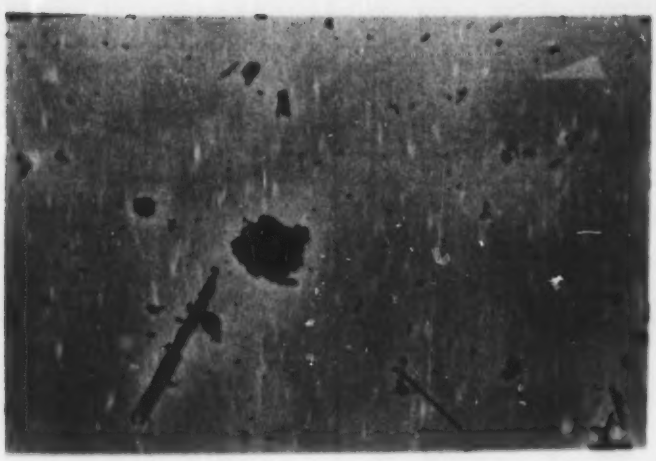
Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	2 (15.4%)	1 (7.7%)	2 (15.4%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	5 (38.5%)
0.12-0.25		1 (7.7%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	1 (7.7%)
0.25-0.50			2 (15.4%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	2 (15.4%)
0.50-1.00				0 (0.0%)	1 (7.7%)	0 (0.0%)	1 (7.7%)	0 (0.0%)	2 (15.4%)
1.00-1.50					1 (7.7%)	0 (0.0%)	1 (7.7%)	0 (0.0%)	2 (15.4%)
1.50-2.50					0 (0.0%)	0 (0.0%)	1 (7.7%)	0 (0.0%)	1 (7.7%)
>2.50						0 (0.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)
Total Fibers	2 (15.4%) [2.0%]	2 (15.4%) [2.0%]	4 (30.8%) [4.1%]	0 (0.0%) [0.0%]	2 (15.4%) [2.0%]	0 (0.0%) [0.0%]	3 (23.1%) [3.1%]	0 (0.0%) [0.0%]	13 (100.1%) [13.2%]
Non-Fibrous	60 [61.2%]	15 [15.3%]	6 [6.1%]	3 [3.1%]	1 [1.0%]	0 [0.0%]	0 [0.0%]	0 [0.0%]	85 [86.7%]
Total Particles	62 [63.2%]	17 [17.3%]	10 [10.2%]	3 [3.1%]	3 [3.0%]	0 [0.0%]	3 [3.1%]	0 [0.0%]	98 [99.9%]

(g) Sample 14 -- Fiber Distribution

Diameter (μm)	Length (μm)								
	≤0.38	0.38-0.75	0.75-1.50	1.50-3.00	3.00-7.50	7.50-15.00	15.00-30.00	>30.00	Total
<0.12	3 (3.1%)	5 (5.1%)	9 (9.2%)	0 (0.0%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	0 (0.0%)	18 (18.4%)
0.12-0.25		0 (0.0%)	14 (14.3%)	1 (1.0%)	0 (0.0%)	1 (1.0%)	0 (0.0%)	0 (0.0%)	16 (16.3%)
0.25-0.50			6 (6.1%)	10 (10.2%)	9 (9.2%)	2 (2.0%)	1 (1.0%)	0 (0.0%)	28 (28.5%)
0.50-1.00				2 (2.0%)	13 (13.3%)	5 (5.1%)	5 (5.1%)	0 (0.0%)	25 (25.5%)
1.00-1.50					2 (2.0%)	5 (5.1%)	0 (0.0%)	1 (1.0%)	8 (8.1%)
1.50-2.50						2 (2.0%)	0 (0.0%)	0 (0.0%)	2 (2.0%)
>2.50						1 (1.0%)	0 (0.0%)	0 (0.0%)	1 (1.0%)
Total Fibers	3 (3.1%)	5 (5.1%)	29 (29.6%)	13 (13.2%)	25 (25.5%)	16 (16.2%)	6 (6.1%)	1 (1.0%)	98 (99.9%)
Non-Fibrous									
Total Particles									

(a) Unmilled Ground & Sieved Material

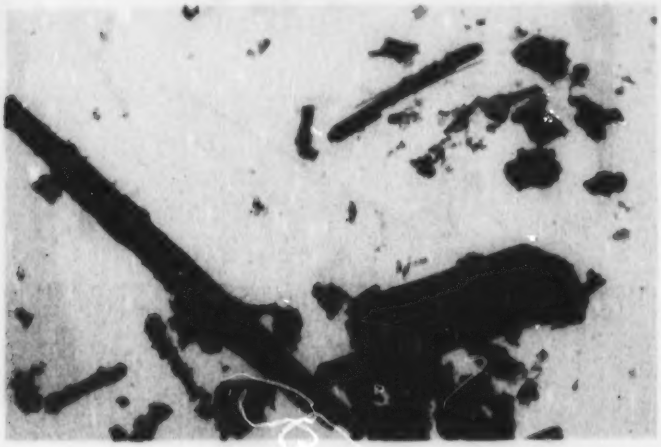
Sample #1 (1000X)



Sample #1 (3300X)

Figure 4. Electron microscope photomicrographs

(a) Unmilled Ground & Sieved Material (cont)



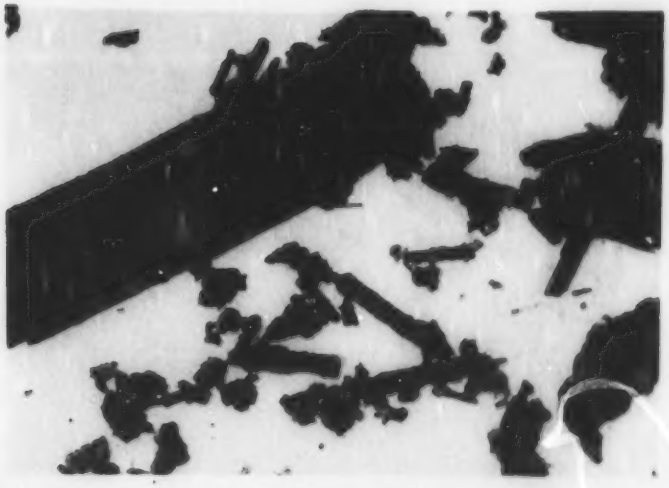
Sample #1 (5000X)

Sample #1 (10,000X)

Figure 4. (continued)

(b) Milled Ground & Sieved Material

Sample #12 (1000X)

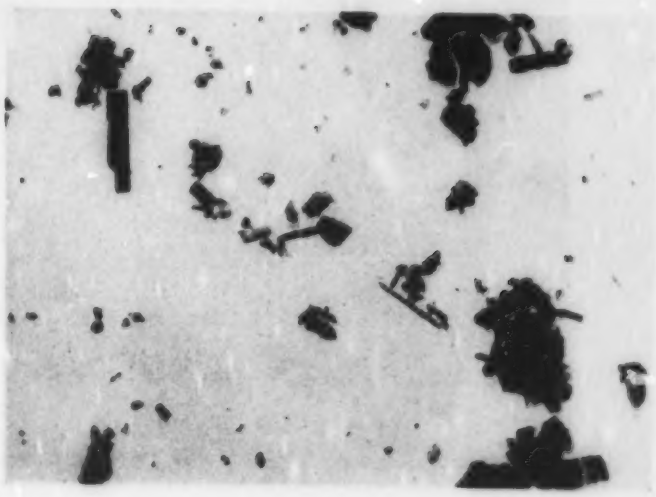


Sample #12 (5000X)

Figure 4. (continued)

(b) Milled Ground & Sieved Material

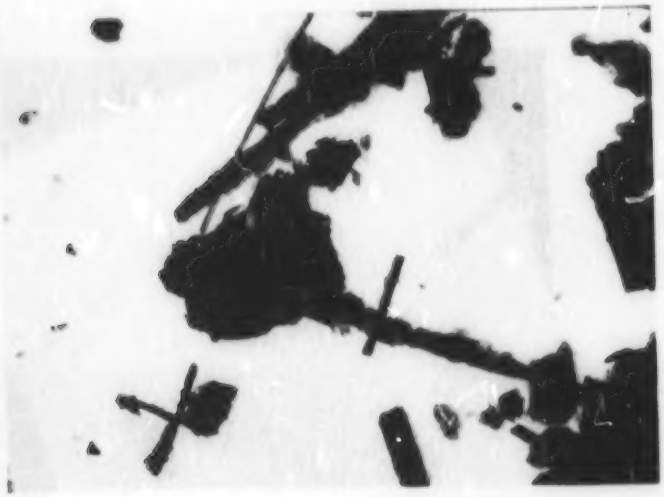
Sample #14 (1000X)



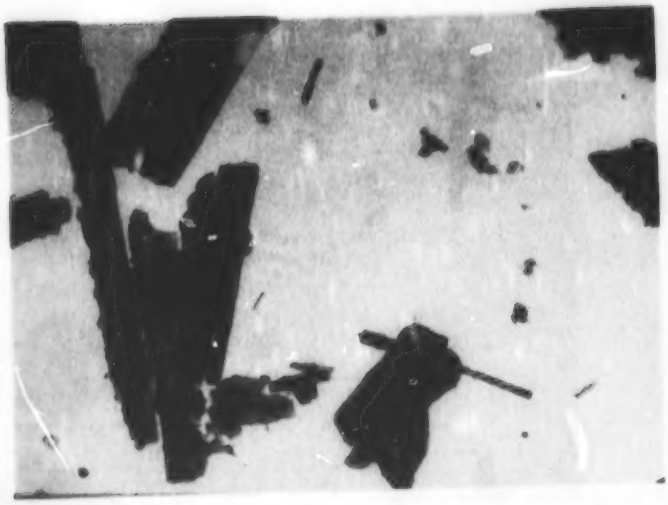
Sample #14 (5000X)

Figure 4. (continued)

(b) Milled Ground & Sieved Material (cont)



Sample #14 (10,000X)



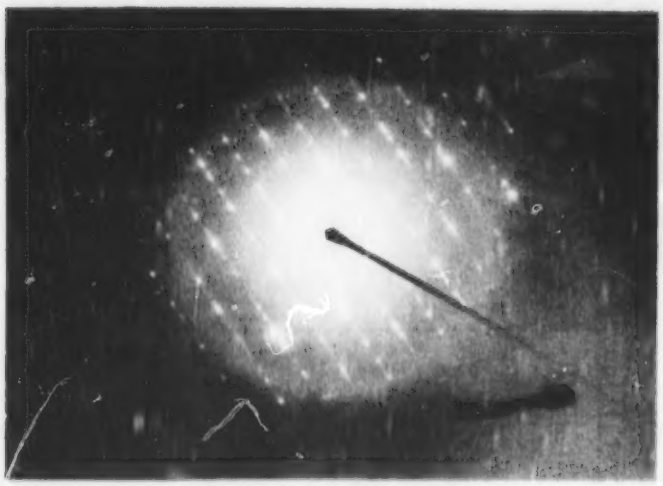
Sample #14 (10,000X)

Figure 4. (continued)

Table 7. ELECTRON DIFFRACTION AND X-RAY SPECTROSCOPY OF PARTICLES

Sample No.	Type of Particle	Number of Particles										
		Crystalline	Non-Crystalline	Total	Mg	Al	Si	K	Ca	Mn	Fe	Total
1	Fibrous	9	0	9	8	0	10	0	6	0	10	10
	Non-Fibrous	9	1	10	4	2	7	1	3	0	6	11
2	Fibrous	11	0	11	7	0	11	0	6	4	11	11
	Non-Fibrous	10	0	10	6	2	9	2	3	1	6	10
12	Fibrous	10	0	10	6	0	10	0	7	3	10	10
	Non-Fibrous	11	0	11	7	0	10	0	3	0	10	10
14	Fibrous	11	0	11	10	0	10	0	10	3	10	10
	Non-Fibrous	12	0	12	11	1	11	1	7	4	11	11

(a) Unmilled Fiber (Sample #1; 20,000X)



(b) Unmilled Fiber (Sample #2; 26,000X)

Figure 5. Electron diffraction patterns of ground and sieved material

(c) Milled Particle (Sample #12; 26,000X)

(d) Milled Fiber (Sample #14; 26,000X)

Figure 5. (continued)

X-RAY SPECTROSCOPY

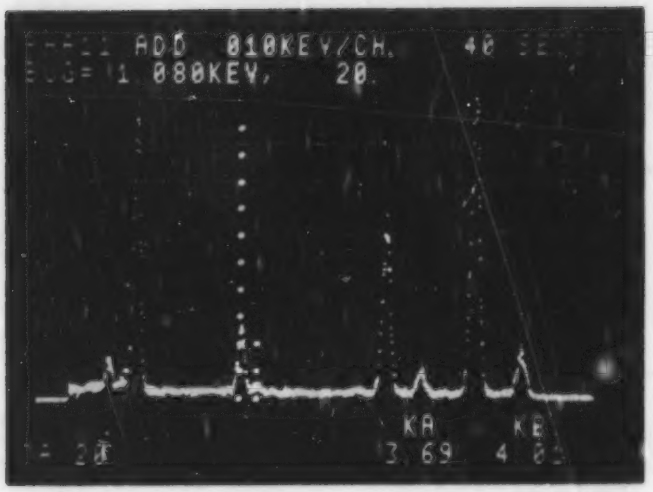
The chemistry of the particles was analyzed by x-ray spectroscopy using the Kevex Energy Dispersive X-ray Spectrometer. Analysis of fibers from fibrous veins and from the ore showed that nearly all of the fibers were silicates with high iron content and with significant amounts of magnesium and calcium (see Table 8). The x-ray spectroscopy results of fibrous and non-fibrous particles in the four samples are given in Table 7. Sample spectra are shown in Figure 6. All of the fibers examined were found to be iron silicates, most of which contained magnesium and calcium and some containing manganese. Most of the non-fibrous particles were iron silicates, many of which contained magnesium and calcium while a few contained aluminum, potassium, and manganese. Sample 14 is again outstanding as all of the fibers examined were silicates containing iron, magnesium, and calcium, while most of the non-fibrous particles examined were similar to the fibers in that all were silicates with iron and magnesium and most contained calcium. The high iron content shows that the fibers are chemically similar to either amosite or crocidolite. However, no aluminum or sodium is present in most of the fibers as they should be in corcidolite.

X-RAY DIFFRACTION

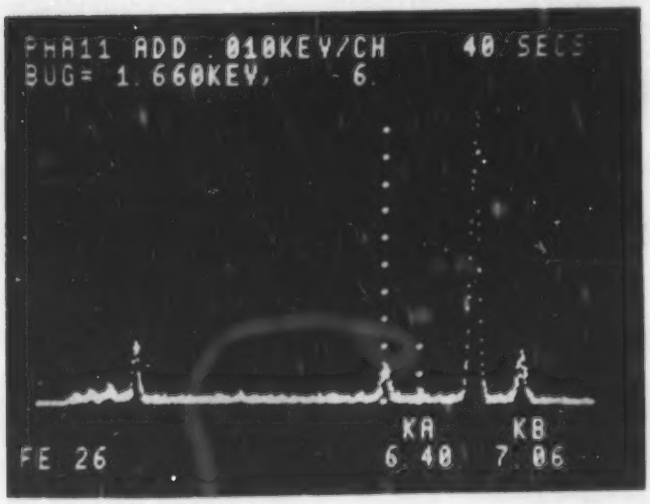
Bulk samples of approximately 5 g were analyzed in the x-ray diffractioner. The results of the analysis for various ore and fibrous samples are given in terms of relative intensities (the intensity of the strongest line = 100) and spacings of the diffraction lines are given in Table 9. The results for the four samples are presented in Table 10.

Table 8. CHEMICAL ANALYSIS BY X-RAY SPECTROSCOPY

Fiber Origin	Percent of Element in Fiber (for 50% Si)					Remarks
	Na	Mg	Si	Ca	Fe	
Chrysotile Standard	2.0-8.0	20-50	50	0.1-3.0	0.5-5.0	
Amosite Standard	0.8-3.0	7.0-9.0	50	0.2-1.5	20-40	
From Fiber Vein	1.4	10.50	50	7.15	23.8	
	1.0	7.90	50	10.60	25.5	
	1.0	8.15	50	10.90	24.4	
	1.0	7.91	50	11.60	26.6	
	0.7	7.55	50	11.40	26.4	
	1.6	8.81	50	11.40	26.7	
	-	6.88	50	11.20	24.8	
	-	8.30	50	11.00	23.9	
	-	11.90	50	9.43	25.0	
	-	-	50	-	31.6	
	3.9	13.60	50	6.62	31.4	
	-	59.80	50	-	-	Non-Amosite
	1.4	4.80	50	11.20	24.8	
	-	6.95	50	12.80	26.6	
	2.7	7.58	50	10.80	24.9	
	-	10.10	50	7.10	47.8	
From Ore	0.9	5.55	50	11.80	25.4	
	3.2	7.55	50	0.60	30.2	
	-	21.40	50	-	20.4	
	-	13.70	50	6.90	24.0	
	2.4	14.90	50	-	24.8	
	-	10.40	50	1.50	26.1	
	-	7.80	50	-	21.0	

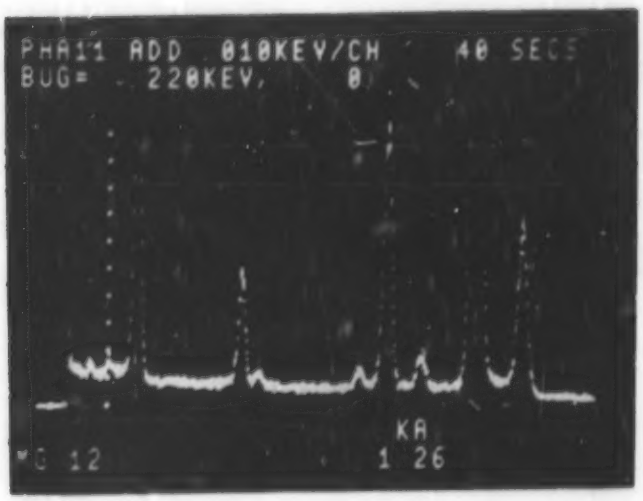


(a) Fiber (0.7x4.0 μm); Sample #1
Mg, Si, Ca, Fe Present

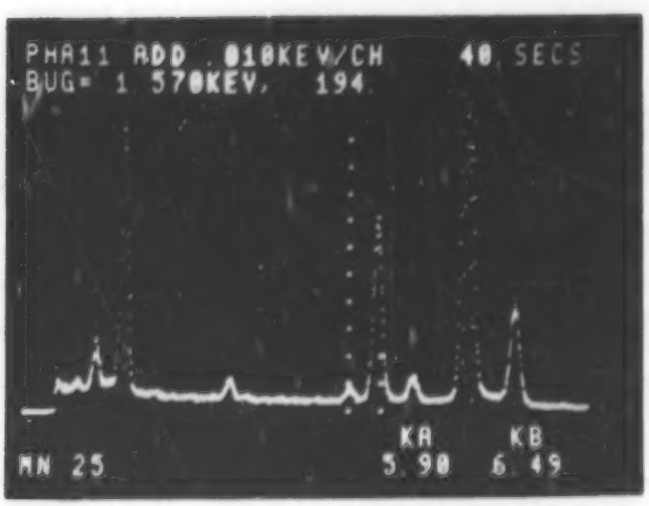


(b) Particle (0.5 μm); Sample #1
Mg, Si, Fe Present

Figure 6. X-ray spectrographs



(c) Fiber (2.0x12.0 μm); Sample #14
Mg, Si, Ca, Mn, Fe



(d) Particle (1.7 μm); Sample #14
Mg, Si, Ca, Mn, Fe

Figure 6. (continued)

Table 9. X-RAY DIFFRACTION LINES PRESENT IN SAMPLES

Hand-Cobbed Fibers		Vibratory-Cobbed Fibers		Fiber-Bearing Rock w/Fibers		Fiber-Bearing Rock w/Fibers ≤ 147 μm		Taconite		Fiber-Bearing Rock w/o Fibers		Cummingtonite-Rich Taconite	
I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)
33	0.84249	57	0.84249							89	0.84089		
						43	0.83456					26	0.83456
				42	0.83065								
		5	0.48216	6	0.48216								
5	0.45753	5	0.45753			15	0.48087			7	0.47958		
										11	0.45521	6	0.45521
						11	0.45063						
												14	0.42704
5	0.42302	5	0.42302					42	0.42402				
						7	0.41520					5	0.39001
										4	0.38667		
												6	0.36776
												4	0.36187
14	0.33264							>100	0.33325			70	0.33448
		5	0.33022			7	0.32903			16	0.33022		
												20	0.32549
36	0.31644	70	0.31534	16	0.31377	37	0.31425	10	0.31865	100	0.31480		
				23	0.30582							50	0.30685
		10	0.29592	23	0.29592	33	0.29497	3	0.29592	7	0.29592		
5	0.28400	10	0.28225	10	0.28139					9	0.28312		
7	0.27303	10	0.27385									12	0.27466
		10	0.27223	10	0.27223	7	0.27143			7	0.27223		
7	0.26180	8	0.26143							9	0.26069	12	0.26217
7	0.25218	18	0.25218	100	0.25218	100	0.25218	7	0.25286	7	0.25355	8	0.25149
								11	0.24551			10	0.24584
		8	0.24169			18	0.24169			7	0.24044		
										7	0.23982		
		5	0.23441	10	0.23500					7	0.23559		
								12	0.22813			6	0.22758
						7	0.22405	9	0.22325				
		5	0.21834	8	0.21885					4	0.21859	6	0.21910
								8	0.21246			6	0.21270
		13	0.20941	35	0.20918	26	0.20895	4	0.20941				
100	0.20490	100	0.20490	52	0.20490	78	0.20490			44	0.20446		
								52	0.20381	44	0.20359	100	0.20381
		5	0.19863	6	0.19852			17	0.19811	4	0.19689	6	0.19852
				10	0.19294					7	0.19160		
5	0.18953	5	0.19065							4	0.19064		
												4	0.18643
		5	0.18344							7	0.18413		
7	0.18022							31	0.18156	7	0.18207	12	0.18173
33	0.17760	40	0.17744	10	0.17744	9	0.17776	13	0.17744	38	0.17744	64	0.17744
								16	0.17680				
				16	0.17098	11	0.17068						
						11	0.16612					4	0.16751
5	0.16556							14	0.16695				
7	0.16327	8	0.16367							4	0.16584	4	0.16584
										4	0.16340		
				42	0.16104	56	0.16104						
		15	0.16078	8	0.16027			3	0.16078				
12	0.15418							22	0.15395			12	0.15395
12	0.15234									9	0.15234		
		13	0.15189							9	0.15144	12	0.15144
		25	0.14796	52	0.14817	52	0.14796	5	0.14817				
		8	0.14547	10	0.14547	7	0.14467	4	0.14507	11	0.14527		
				10	0.14250	11	0.14192						
		5	0.14079	13	0.14079							6	0.14041
								19	0.13786			16	0.13741
								28	0.13733				
						11	0.13440						
						7	0.13021						
								5	0.12854				
						11	0.12780	2	0.12839				
								5	0.12549				
								5	0.12521				
								5	0.12276				
								8	0.11993				
								8	0.11786				
								6	0.11521				

Table 10. X-RAY DIFFRACTION LINES PRESENT IN SAMPLES OF GROUND AND SIEVED FIBROUS ROCK

Sample No. 1 Unmilled		Sample No. 2 Unmilled		Sample No. 12 Jet-Milled		Sample No. 14 Jet-Milled	
I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)
100	0.850	100	0.843	100	0.838	100	0.842
7	0.482					6	0.482
7	0.457	8	0.479	11	0.477		
				9	0.457		
		3	0.452	9	0.452		
5	0.343	8	0.340	4	0.342	10	0.339
9	0.333			4	0.337		
		11	0.331	9	0.330	16	0.330
82	0.315	74	0.315	67	0.314	81	0.315
7	0.296	8	0.295	7	0.295	6	0.296
9	0.284	8	0.282	11	0.282	10	0.282
11	0.273	16	0.273	18	0.273	29	0.273
7	0.262	8	0.262	9	0.262	10	0.262
5	0.255	11	0.255	11	0.254	16	0.255
7	0.240	5	0.242	9	0.240		
9	0.236	8	0.235	9	0.235	13	0.236
9	0.218	8	0.218	11	0.218		
5	0.166	8	0.166			10	0.166
5	0.160						
7	0.148						
7	0.145	11	0.145	7	0.149	10	0.145

Standard X-ray diffraction lines of some inorganic materials and asbestos types are given in Tables 11 and 12. A summary of observations made from these results are given in Table 13. It should be noted that many other materials could be present; there is some inaccuracy in the method; concentration effects may mask the presence of some materials and identification of the materials present is somewhat subjective and only within reasonable accuracy. Cummingtonite or grunerite and amosite or crocidolite may be present in all of the samples analyzed except the typical taconite. The analysis of the four ground and sieved samples are nearly identical. They all appear to contain cummingtonite, riebeckite and richerite with significant trace amounts of tremolite and crocidolite. This suggests that at least the ground and sieved material is structurally more similar to crodidolite than amosite. As no sodium or aluminum was found to be present in the fibers as would be in crocidolite and the amount of calcium was found to be higher than normally found in the grunerite-cummingtonite series of which amosite is member, the results suggest that the fibers are in the tremolite-actinolite series.

ATOMIC ABSORPTION

Atomic absorption analysis was conducted on approximately 0.1-0.2 g amounts of the ground and sieved samples for four trace metals (Mn, Ni, Cr, Co) using a Jarrell-Ash Model 82-528 Maximum Versatility Atomic Absorption-Flame Emission Spectrophotometer. The results are presented in Table 14. Manganese was found to be present in fairly large quantities (on the order of 10 mg per gram of sample) while nickel, chromium, and cobalt were present in smaller quantities. All of these elements are present in trace quantities in many types of asbestos.

Table 11. X-RAY DIFFRACTION LINES IN SOME INORGANIC MINERALS

Nickel Holder		Quartz		Grunerite		Cummingtonite		Riebeckite		Tremolite		Richerite		Fayalite		Paragonite Basic Na-Al-Silicate		Al-Montmorillonite Basic Al-Silicate	
I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)
						70	0.838	100	0.840	100	0.838								
				100	0.833														
										•									
				30	0.484					10	0.487								
				30	0.468					20	0.476								
				35	0.458														
		35	0.426					20	0.451	20	0.451						0.439		
										35	0.420								
				40	0.416														
				50	0.383					16	0.387								
										40	0.378								
		100	0.334	55	0.347			16	0.342				0.338						
				50	0.328	60	0.326			75	0.327								
								55	0.311	100	0.312		0.315				0.318		0.317
				80	0.307	80	0.307			10	0.303						0.303		
				40	0.300	60	0.299	20	0.298	40	0.294		0.294						
										45	0.280				0.282				
				90	0.277	100	0.275	50	0.273	16	0.273								
										90	0.270		0.271						
						60	0.262			30	0.259								
				60	0.251	60	0.251	45	0.253	40	0.253		0.253		0.250		0.251		
			0.246																
				30	0.241					8	0.241						0.241		
										30	0.238								
								16	0.232										
			0.228	40	0.230	40	0.229												
			0.224					10	0.226										
						40	0.218	10	0.218				0.216						
			0.213																
																	0.208		
100	0.203					30	0.203												
			0.198																0.199
																			0.186
		17	0.182																
															0.178				
42	0.176																		
			0.167																
						40	0.165												
			0.154	20	0.153														
				15	0.152														
				10	0.151														
				10	0.148												0.148		
				35	0.141	40	0.140												
			0.138	25	0.139														
			0.137	20	0.137														
				10	0.133														
				35	0.131														
			0.129																
				10	0.128														
			0.126																
21	0.125																		
			0.123																
			0.120	20	0.119														
			0.115																

Table 12. X-RAY DIFFRACTION LINES IN MAIN ASBESTOS TYPES

Amosite		Anthophyllite		Crocidolite		Rhodesian Chrysotile		Canadian Chrysotile	
I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)	I	d(nm)
		100	0.950	100	0.843				
		26	0.840						
100	0.826					100	0.738	100	0.738
		56	0.458						
				26	0.451	29	0.455	27	0.455
						51	0.366	57	0.366
				26	0.343				
36	0.327	47	0.325						
		58	0.313	41	0.311				
52	0.307	64	0.306						
43	0.277								
				50	0.272				
				20	0.261				
				36	0.254	27	0.246	24	0.246
						20	0.154	21	0.154

Table 13. SUMMARY OF X-RAY DIFFRACTION RESULTS

Mineral	Sample										
	Hand-Cobbed Fibers	Vibratory-Cobbed Fibers	Fiber-Bearing Rock w/Fibers	Fiber-Bearing Rock w/Fibers ≤ 147 μm	Taconite	Fiber-Bearing Rock w/o Fibers	Cummingtonite-Rich Taconite	#1	#2	#12	#14
Quartz	T	M	N	M	P	M	P	N	N	N	N
Grunerite	N	N	M	T	N	M	P	M	M	M	M
Cummingtonite	M	T	M	P	N	M	P	P	P	P	P
Riebeckite	T	M	M	T	N	T	N	P	P	P	P
Tremolite	N	M	N	M	N	M	M	T	T	T	T
Richerite	M	P	M	T	M	M	N	P	P	P	P
Fayalite	M	M	M	M	M	M	M	M	M	M	M
Paragonite (Basic Na-Al-Silicate)	N	M	M	M	M	M	N	M	M	M	M
Al-Montmorillonite (Basic Al-Silicate)	M	M	M	M	M	M	M	N	N	N	N
Amosite	M	M	T	M	M	M	M	M	M	M	M
Anthophyllite	M	M	N	M	N	M	T	M	M	M	M
Crocidolite	T	T	N	M	N	M	M	T	T	T	T
Rhodesian Chrysotile	N	N	N	N	N	N	M	N	N	N	N
Canadian Chrysotile	N	N	N	N	N	N	M	N	N	N	N

P - Definitely Present

T - Present as Trace Material

M - May be Present

N - Not Present

Table 14. ATOMIC ABSORPTION

Sample No.	Sample Wt (g)	Weight (µg)				Metal Wt/Sample Wt (µg/g)			
		Mn	Ni	Cr	Co	Mn	Ni	Cr	Co
#1	0.1464	1900	37.5	6.0	3.75	12980	256.1	40.98	25.61
#12	0.1140	1650	50.0	4.5	3.00	16670	438.6	39.47	26.32
#14	0.1337	1500	30.0	4.5	3.00	11220	224.4	33.66	22.44

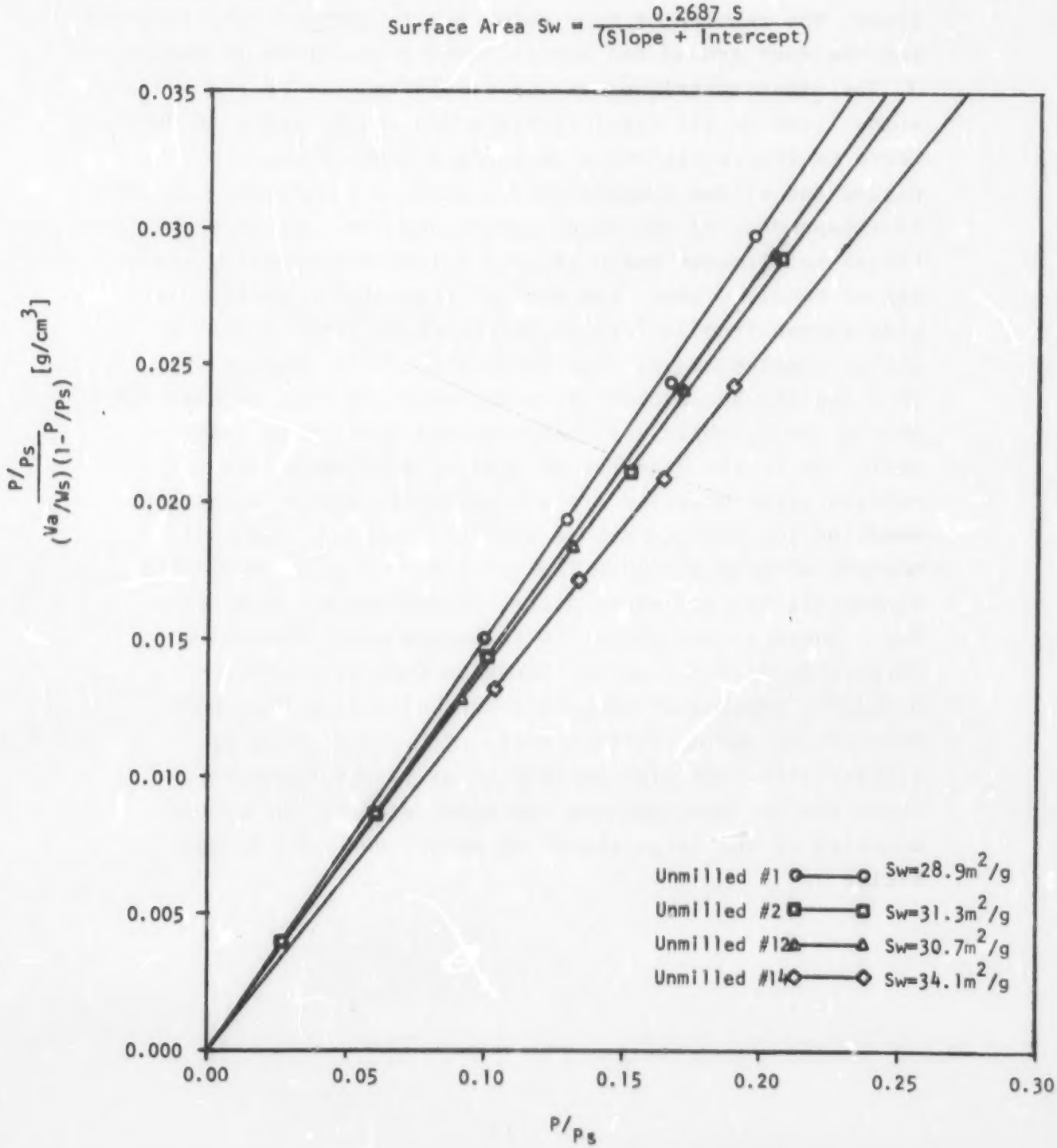
Table 15. BENZENE EXTRACTION OF ORGANICS

Sample No.	Sample Wt (g)	Weight Organics (mg)	Organics Wt/Sample Wt (mg/g)
#1	0.4933	1.0	2.03
#12	0.4035	1.7	4.21
#14	0.4503	1.0	2.22

B.E.T. SURFACE AREA

B.E.T. Surface Area Analysis was conducted using a Micromeritics Model 2100D Orr Surface-Area Pore-Volume Analyzer. The results of four point B.E.T. surface area analysis for the four ground and sieved samples are shown in Figure 7. The experimental points lie almost exactly on the regression lines in all cases (correlation coefficients ≥ 0.9999). There is little variation in surface area between the unmilled and milled samples as the surface area-to-volume ratio is independent of the fiber length and inversely proportional to diameter which is only slightly reduced by breaking of bundles. Thus, the surface area of the milled samples ranges from 30.7 to 34.1m^2/g, while that of the unmilled samples ranges from 28.9 to 31.3m^2/g showing that only bundles are broken up to increase the area exposed for absorption slightly and that they are reduced in length while not in the diameter of individual fibers. The B.E.T. surface areas measured are significantly higher than those measured for UICC amphibole asbestos samples. Timbrell[16] measured values of 5.7 ± 0.3, 11.8 ± 1.0, $8.3 \pm 0.5 m^2/g$ for amosite, anthophyllite, and crocidolite, respectively. Chrysotile has a surface area closer to those measured (Rhodesian Chrysotile $21.3 \pm 1.5 m^2/g$; Canadian Chrysotile $26.8 \pm 0.7 g/m^2$). Samples of tremolite and actinolite have been measured as having surface areas of 6.2 and 11.6m^2/g, respectively. The high surface areas values obtained for these samples may indicate the small size of the ground material or the large amount of small, non-fibrous particles.

Figure 7. B.E.T. surface area analysis



BENZENE EXTRACTION

Soxlet extraction of benzene soluble oils was conducted for at least four hours. The results are shown in Table 15. The values of oil content measured ranged from 2.0 to 4.2 mg/g of sample. These values are approximately fifty times greater than those measured for UICC samples of amosite, anthophyllite, and crocidolite, and approximately ten times greater than those measured for UICC chrysotile[16].

SECTION 8

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